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BUREAU OF MINES
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ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL FISCAL YEAR 1946

BY

A. C. FIELDNER AND P. M. AMBROSE

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ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL

Fiscal Year 1946[1]

By A. C. Fieldner[2] and P. M. Ambrose[3]

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INTRODUCTION

This, the 11th annual report of research and technologic work conducted by the Bureau of Mines on coal and coal products, is issued following the closing of the Bureau's war work on fuels and contains references to most of the published articles containing results of war investigations. The few remaining papers will be discussed in next year's report. The trend in coal research in the Bureau of Mines was discussed in Mechanization.[4/] This publication, covering the period from the close of armed conflict to resumption of normal coal-research activities, includes a section never previously presented. There is included for the first time a discussion of the Bureau's activities in the Liquid Fuels and Lubricants and the Solid Fuels Subcommittees of the Technical Industrial Intelligence Committee. This report does not replace the more comprehensive ones prepared

4/ Fieldner, Arno C., Coal Research for 1946: Mechanization, February 1946, pp. 105-109.

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by individual authors and listed as footnote references in this publication. Its purpose is to bring to the reader a résumé of the work for the fiscal year ended June 30, 1946. The material presented herein is based largely upon publications, but the results of some research and testing, which would not otherwise be published in the near future, are presented in detail.

SUMMARY

The Bureau of Mines continued its studies and services for increasing the economy and efficiency in governmental, industrial, and domestic fuel-using operations. Analytical-testing programs were designed to develop substitutes to overcome the depletion of high-grade coal resources, and process development went forward to utilize advantageously the great lignite deposits of the Northwest.

Services to Federal agencies included the sampling and analysis of coal and tests of fuels and boiler-room equipment. Analyses of boiler water and recommendations on fuel purchasing and boiler-water treatment were made. The coal-analysis laboratory examined a total of 15,601 samples. Of these, 5,910 were samples of coal-mine dusts taken as part of the safety program for the reduction of dust explosions, 4,674 were samples of coal taken in connection with Government purchase and tipple and breaker inspections, and 5,017 were samples of coal and related materials examined for research and related groups. Types of fuel and fuel-burning equipment were determined for 37 new hospitals for the Veterans Administration. Consulting service on smoke-abatement work was given to a number of cities.

Five engineers of the Bureau of Mines were detailed to the Foreign Economic Administration to serve under the Technical Industrial Intelligence Committee to obtain the latest hitherto unavailable information on European practice in the mining, preparation, and utilization of coal. Another was detailed to obtain information in Japan on low-temperature carbonization of coal.

With the close of the war, the National Fuel Efficiency Program was terminated. Cooperation of 16,000 industrial organizations had been obtained, and the estimated savings had reached the rate of 5,000,000 tons of coal amounting to some $30,000,000 per year.

The construction of a new-type scraper-shaker-gangway loading machine is virtually complete, and tentative arrangements have been made to test this equipment and special shearing machines in mining thin, steeply pitching beds of anthracite. A study of utilization of light earth-moving equipment in bituminous-coal strip mining was completed. The development of a coal planer uncovered in Europe has been arranged. Five million tons of lignite minable at low cost by strip-mining methods were proven in a deposit in Washington. Exploration for coking coal, critically needed for long-term, economically competitive operation of the western steel industry, was continued in Colorado. Preliminary results of coking tests indicated coal equal to the best previously known western coking coal. Exploration was also conducted in Alabama for coking coal and in Maryland for highly desirable semi-smokeless coal of coking quality and in Alaska for coal necessary for regional

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use. More than 1,800 feet of drill cores from coal beds at seven exploration projects were studied in detail, and nearly 1,000 feet of coal from drilling received petrologic examination, chemical analysis, and other tests.

Studies were made of coal seams in 11 States to determine their adaptability to the production of a very low-ash washed coal for the production of synthetic liquid fuels by the hydrogenation process. It was shown that some strip coal in the State of Washington, otherwise never mined, could be made marketable by washing. A study of the preparation characteristics of the thinner coal seams in the State of Maryland was undertaken, aimed at the development of these resources as the more important beds become depleted. A similar project has been initiated in Alabama as a part of a general drilling and exploration program. Assistance has been given Alaska and several South American countries to enable them to utilize their coal resources better through preparatory treatment. Improved preparation practice, combined with the use of the ash-removal stoker, has broadened the markets for high-ash coals with high-fusion-point ash.

Four years of study, involving the use of 30,000 tons of coal, proved that subbituminous coal could be stored without danger of spontaneous heating and little loss in heating value by using simple methods to prevent air circulation within the storage pile. Advice on storage facilities and the storing of particular coals was given to the many Federal fuel-consuming facilities upon request. Investigation was begun of a new process, developed in Holland, for cleaning fine coal. Recoverable montan wax (hitherto imported) has been found in lignite from Malvern, Ark., and Ione, Calif.

An investigation designed to minimize or prevent corrosion of boiler tubes has led to a procedure for evaluating various protective tube coatings. To obtain more efficient boiler design, studies have been made of the heat absorption in large central-station boiler furnaces. In connection therewith, the examination of the properties of coal-ash slag was continued. Slag properties are of interest in the utilization of Rhode Island anthracite in a slagging-gas producer designed to make rock wool as well as gas. To evaluate fuels from new processes and packaged fuels from various sources, using various binders, a test procedure has been developed.

To offset the depletion of the best known coking coals under war and reconstruction demands, the survey of carbonization properties was continued. Examinations included the expanding, plastic, and oxidation properties, particularly as related to the adaption of various coals in the making of metallurgical coke. Laboratory tests indicated that Beckley-bed coal from West Virginia might be suitable for blending with high-volatile coal in coking mixtures. Coking and byproduct tests were made on three coals from Colorado and four from West Virginia, as well as several South American coals for the State Department.

Studies on the utilization of lignite showed that gas suitable for the reduction of iron ore or gas suitable for the synthesis of liquid fuels could be made in externally heated alloy retorts.

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Investigations on synthetic liquid fuels are carried out under Public Law 290 (78th Cong.) to develop processes for making gasoline and oil from coal, oil shale, and other substances and apply them on a demonstration-plant scale. This legislation was passed, funds were provided, and the work was started as the demand for oil products rose to unprecedented heights during the war and discoveries of oil in new fields in this country fell to about one-third of the annual requirements. The great war demand for oil no longer exists, but, contrary to expectations, the country's normal demand is now at about the same level as the peak reached during the war, and results from exploration are no better. These conditions indicate that the need for the synthetic-fuel work is greater now than it was at the time the legislation was passed and that it will steadily become more important as the country's industrial activity increases.

Two processes are being studied on the research and development scale for production of oil and gasoline from coal. This work is in Pittsburgh, Pa., the nearby town of Bruceton, and Morganton, W. Va. Equipment now in operation provides for the production and testing of catalysts, preparation of coal, gases, and other raw materials for the processes, and the operation of two complete units of equipment for the continuous synthesis of oil and gasoline from coal. Other synthesis units, producing up to about a barrel of oil a day, are being completed and will be in operation as soon as laboratory space is available in new buildings that are being erected and will be available this fall.

Significant progress has been made in one of the processes, both in preparation of the catalysts and in removal of the heat of reaction, which has been one of the bottlenecks in the design and construction of large units of commercial equipment. It is estimated that by this process these improvements can cut production costs in half. Similar possibilities for cutting costs are in sight for the second process for making oil from coal.

A demonstration plant to produce 200 barrels of oil a day by the hydrogenation of coal is being designed for erection at Louisiana, Mo., on the site of the Government-owned synthetic-ammonia plant located there. It is expected this plant will incorporate the major features of design worked out in the development program.

Tests of new permissible explosives, permissible samples collected in the field, and new types of industrial explosives seeking to utilize surplus military stores were made. Two new explosives were added to the permissible list, which now includes 180.

An extensive investigation on the hazards of toxic gases from sheathed explosives established the limitations of such explosives and the conditions under which they may be employed. Industrial cooperative studies were made on the performance and hazards of liquid-oxygen explosives, which will be of material aid to field operators.

Significant advances have been made in the accurate determination of hazards of static electricity produced by friction or successive contact

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and separation of unlike surfaces, in the manufacture, storage, and use of explosives and inflammable substances. The precise measurement of the electrostatic charge acquired by personnel and equipment during typical operations enabled setting up safety standards.

Study of the ignition of firedamp by explosives revealed that the geometry of the explosive and the position of the detonator relative to the charge play an important role in the ignition process.

Special inquiries from industry and other Government agencies brought about investigations of the use of hydrogen peroxide as an oxidizing agent in explosives, in the evaluation of hazards in specific industrial operations, and in devising methods of measuring rates of detonation with a high degree of precision.

Further tests in the experimental coal mine connected with the safe use of larger explosive charges have confirmed the desirability of continuing the 3-pound explosive-charge limit for coal mining in lieu of the previous 1-1/2-pound limit.

Knowledge of the mechanism of ignition of explosive gas mixtures by electric sparks has been advanced by the accurate determination of minimum spark energies for ignition.

Fundamental studies of flame have developed principles governing the performance of gas burners that will aid in the scientific designing of burners, including jet burners of all types.

For the first time, research has made it possible to state conditions for eliminating explosions in medium-pressure acetylene generators by adding prescribed amounts of hydrocarbons such as natural gas, propane, or butane to acetylene as it is produced in the generator. Solid and gaseous products of unusual interest were found to result from explosion of such mixtures. Other investigations of combustible materials involved determination of minimum ignition temperatures, limits of inflammability, prevention of gasoline and naphthalene explosions and the use of helium or Freon in explosion prevention.

Over 60 samples of industrial dusts and powders for special purposes submitted by industry, some of which had already caused explosions and fires, were studied for their hazardousness, and recommendations for preventing such explosions were formulated.

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This report includes work done under the technical direction of the following members of the staff of the Bureau of Mines:

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University of Washington

ORIGIN, COMPOSITION, AND PROPERTIES OF COALS

Inspection, Sampling, and Analysis

A shortage of coal existed throughout the year, and, in general, Government agencies had difficulty in obtaining enough coal to meet requirements. The Office of Indian Affairs, the Department of Justice, and, to a limited extent, the War Department purchased coal on a guaranteed-analysis basis. The rest was purchased on a flat-price basis and in some instances was obtained through directives by the Solid Fuels Administration for War. Consulting service, including in some instances the analysis of bids and recommendations of awards and recommendations on coal specifications, was given the War Department, Navy Department, Department of Justice, Post Office Department, Procurement Division, Treasury Department, and Department of the Interior. Coal purchases by Federal Agencies during the fiscal year were originally estimated at 10,000,000 tons, the War Department being the largest purchaser and the Navy Department the second largest. Many War Department posts were closed during the year, resulting in a decrease in the tonnage actually purchased.

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Analyses were furnished all purchasing agencies for use in evaluating coal bids and for use when substitute coals were offered. In connection with contracts for furnishing this coal, 4,672 reports on proximate or ultimate analysis and heating value of coal and 1,893 reports on fusing temperature of ash were issued to Federal Agencies. These reports provided analyses for price adjustment where applicable and for other action as provided by the contracts, such as cancellation of contracts, refusal of further shipments, and rejections of coal.

Coal sampling at Army posts, begun in September 1943 upon request of the War Department, was continued, and particular attention was given to instructing Army personnel in proper methods of sampling. In all, 1,442 visits were made to Army posts and 1,343 samples were collected. Upon the request of the War Department, coal loaded for export was inspected and sampled at the posts of Pensacola, Fla., Mobile, Ala., and Charleston, S. C., and, to determine suitability of coal for export, inspections were made at 18 mines in Alabama.

Sampling at Pennsylvania anthracite breakers, begun in November 1943 upon the request of the Solid Fuels Administration for War, was continued, and 144 samples were collected at 40 breakers. As a result of this work, a number of breakers were shut down, others installed new cleaning equipment, and in general the quality of anthracite shipped was improved. Upon the request of the Office of Price Administration, 69 samples of Pennsylvania anthracite were collected at railroad weigh scales for the purpose of providing data upon which to base price adjustments for substandard coal.

Visits were made to 29 agencies of the Indian Service for the purpose of checking sampling methods and instructing Indian Service personnel in proper sampling methods. Samples of coal were collected for special purposes in various States. The coals included 38 face samples and 11 five hundred-pound samples at 11 mines in Alabama to determine suitability of the coals for use in a Lurgi pressure-type gas generator; 7 samples ranging from 250 to 1,000 pounds for hydrogenation tests; and 5 samples for microscopic examination at 6 mines in Illinois; two 1,000-pound samples for hydrogenation tests and two for microscopic examination at 2 mines in Missouri; 65 samples at 60 mines in Utah, 30 samples at 22 mines in Wyoming, and 1 sample at 1 mine in Idaho for preliminary carbonization tests at the Golden station; 1 sample at a mine in Illinois and 1 at a mine in Colorado for tests on prolonged air-drying loss; 2 samples at 2 mines of Pennsylvania semianthracite for microscopic examination; and the following samples for analysis for the general use of Federal Agencies in determining awards of contracts and for information for public use: 62 tipple samples at 21 mines in West Virginia, 1 tipple sample at 1 mine in Pennsylvania, 6 tipple samples at 2 mines in Ohio, 54 tipple samples at 16 mines in Illinois, 1 tipple sample at 1 mine in Colorado, 15 tipple samples at 3 mines in Montana, 81 tipples at 29 mines and 4 face samples at 4 mines in Wyoming, 149 tipples at 56 mines and 38 face samples at 10 mines in Utah, and 3 tipple samples at 1 mine in Idaho.

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Analyses of Alaska Coals

A compilation[5] of analyses of Alaska coal was published and is another in the series of technical papers describing coals of individual producing States. The geology of the coal fields, production, distribution, uses, and the relationship of mine samples to commercial shipments were discussed. Descriptions of mine and delivered samples included chemical analyses, calorific values, classification by rank, agglomerating indices, and fusibility of ash temperatures. Descriptions were given of the location, thickness of bed, nature of floor and roof, and partings of a number of operating mines. A map of Alaska showing principal areas known to contain coal was included.

Data on Coals in District No. 9, Western Kentucky

A data book[6] giving information on coal produced in western Kentucky (Producing District No. 9) was published and is another in the series heretofore prepared by the Office of the Bituminous Coal Consumers' Counsel in collaboration with the Bureau of Mines. The book contained descriptive tables of direct rail-connected and ramp-loading mines, with seam names, mine index numbers, and originating railroad or river, maps showing their locations, and tables giving specimen analyses (with usual ranges) by seams and counties.

The number of samples analyzed in connection with Government fuel purchases declined slightly owing to reduction in force of several samplers after the termination of hostilities. Samples analyzed for the War and Navy Departments accounted for 74 percent of the Federal samples. Research of the Bureau on exploration, preparation, and utilization of coal and coal products required analysis of 5,017 samples. These samples came from 16 States, Alaska, Chile, China, Ireland, Italy, and Peru.

Assistance was given to Federal coal-mine inspectors by analyses of 5,910 samples of coal mine-dust collected on the ribs, roofs, and floors of 904 coal mines in 18 States. These analyses were used in determining whether corrective recommendations were necessary at specific mines and in formulating the recommendations where necessary.

In addition to analytical services given to the several sections of the Bureau of Mines and other Federal Agencies in analyzing 15,601 samples requiring 164,533 determinations, portions of samples recently analyzed were sent to 14 coal laboratories or schools for the purpose of checking equipment and for student use.

5/ Fieldner, A. C., Gates, George O., Bell, C. H., Anderson, R. L., Snyder, N. H., Cooper, H. M., Abernethy, R. F., Tarpley, E. C., and Swingle, R. J., Analyses of Alaska Coals: Bureau of Mines Tech. Paper 682, 1946, 114 pp.

6/ Fieldner, A. C., Sweeny, Harry P., Rice, W. E., and Moran, H. E., Typical Analyses of Bituminous Coals Produced in District 9: Bureau of Mines Data Book, vol. 6, 1945, 32 pp.

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Constitution, Properties, and Analytical Methods

Extractable Waxes in United States Lignites

Solvent extraction of certain German brown coals with either benzene or a mixture of benzene and alcohol yields an extract that is termed montan wax. The montan-wax industry prior to the war had been developed extensively in Germany, as the wax finds use in many industrial applications. In 1939, over 7,000,000 pounds of montan wax was imported into the United States, nearly all of which was shipped from Germany. As little information on the potential use of American low-rank coals as a source of wax is available, an investigation is in progress to determine the nature and amount of material extractable from such coals.

Preliminary extractions of dry lignite with benzene as solvent have been made on samples from deposits in Arkansas, California, North Dakota, Texas, and Washington. The results show that a considerably greater quantity of extract was obtained from the samples examined from Ouachita County, Ark., and Amador County, Calif., than from the other sources. The Arkansas and California lignites gave 6 to 7 percent of extract on a dry-coal basis, whereas the North Dakota, Texas, and Washington samples usually yielded less than 1 percent of extract. Slightly higher yields were obtained from air-dried samples than from samples dried at 105°C. If an azeotropic mixture of benzene and alcohol is used as solvent, considerably higher yields of extract are obtained from air-dried samples than if benzene is used alone. Yields of extract from the Arkansas and California lignites are as high as 11 to 13 percent on a dry-coal basis using the benzene-alcohol mixture as solvent. The properties of the wax extracted from a sample of California lignite is dependent somewhat on the solvent used in the extraction. The acid number and saponification number both increase with increase in the amount of extract obtained. The melting point, ester number, and so-called "resin" content show only small differences.

Although a number of tests have been used for evaluating montan wax, those most quoted in the literature are the melting point, acid number, saponification number, ester number, and "resin" content. The term "resin" is usually applied to those constituents that are more soluble in solvents as ethyl ether, ethyl acetate, ethyl alcohol, and benzene-alcohol mixtures at or below room temperature than is the true ester wax. The results that have been completed indicate that the waxes from the Arkansas and California lignites examined have properties similar in many respects to crude montan wax from German brown coal. One difference so far detected is that the waxes examined may have a higher "resin" content than crude commercial montan wax.

Analyses of Miscellaneous Materials

A total of 195 samples of miscellaneous materials was analyzed and reported during the year. The samples represented a wide variety of materials for which chemical analyses or tests were requested in connection with investigations of coal utilization or of problems concerned with health and safety.

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Forty samples, including 25 catalysts, were analyzed in connection with the Synthetic Liquid Fuels Program. Twenty-four of the catalysts were collected from German synthetic liquid fuel plants and were analyzed to identify the active constituents. Molybdenum was determined in a molybdenum catalyst and in three catalyst residues from a converter. Tin was determined in a residue from coal hydrogenation where tin sulfide was used as a catalyst. Water from a gas holder was tested for sulfur, and seven samples of kieselguhr, distilled water, water conditioner, and magnesium oxide used in preparing catalysts for Fischer-Tropsch synthesis were examined for impurities.

Seventeen samples of coal were analyzed for water-soluble chloride as a part of a study of the preparation of coal adaptable for use in coal hydrogenation. On an air-dried coal basis, the amount of chloride calculated as Cl ranged from 0 percent each for a Montana and a North Dakota coal to 0.36 percent for an Indiana coal. Samples of ash from another series of 23 samples of coal were analyzed for SiO_2, Fe_2O_3, CaO, and Mn_3O_4. Fifteen of the ash samples represented 1-1/2-inch by 14-mesh coal mines in Illinois, Indiana, Kentucky, Montana, North Dakota, Ohio, Pennsylvania, Utah, West Virginia, and Wyoming. Eight samples represented float on 1.60 specific gravity of 3/8-inch by 14-mesh coal from eight of the same mines. Fusain in two samples of coal was determined by a chemical oxidation method. A sample of minus 5/8-inch raw screenings from Illinois No. 6 bed coal contained 5.0 percent fusain, and a channel sample of Pittsburgh-bed coal from the Experimental Mine contained 4.2 percent. In comparison, relatively inert organic material determined petrographically by the powdered-coal method in the same samples was 4.4 and 5.0 percent, respectively.

A total of 50 analyses was made in connection with fuel engineering service of the Bureau of Mines to other Government agencies. These samples included 11 boiler-water compounds, 2 samples of tannin used in boiler-water treatment, 16 boiler scales and deposits, and 2 boiler waters analyzed in connection with work on boiler feed-water conditioning in Federal power plants. Also, 3 external deposits on boiler tubes, 5 fly ashes, and 11 deposits on air-preheater plates of steam plants were analyzed as part of investigations of corrosive deposits on boiler surfaces.

Nineteen samples of slag, residue, coke ash, and limestone were analyzed in connection with tests of a slagging-gas producer using Rhode Island anthracite for the direct production of mineral wool.

Nine samples of petroleum coke contained 0.02 to 0.05 percent silicon and 0.05 to 0.09 percent iron. Three samples of foundry coke contained 0.008 to 0.023 percent phosphorus, which is considerably under the maximum of 0.09 percent permitted in the specifications. A face sample of coal from Pierce County, Wash., contained 0.08 percent phosphorus. A sublimation product collected from the surface of a smoldering refuse dump at the Cadogan mine, Armstrong County, Pa., contained mainly free sulfur and ammonium chloride. Iron was determined in the ash of lignite, char, blow-over dust, and sludge from a test run at the Bureau of Mines lignite-gasification plant at Grand Forks, N. Dak., to assist in making a materials balance.

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Five samples of limestone and four samples of fly ash were examined to determine their suitability for rock-dusting coal mines to prevent the propagation of coal-dust explosions. The fly-ash samples, representing fine ash from furnaces burning powdered coal, were considered unsuitable for rock dusting because they were highly siliceous and contained 6.3 to 8.4 percent combustible matter, which is more than the maximum of 5 percent allowed in Mine Safety Board Decision 32. Four of the five limestones examined were of sufficient purity to be suitable for rock dusting. Flash and fire points were estimated for a sample of gilsonite submitted by the Michigan Department of Health for tests of explosibility of the dust. A sample of resin removed from an Army Ordnance canister containing steel balls imbedded in resin was tested to determine its softening point, flash point, and fire point as part of a study of the feasibility of using surplus canisters as steel scrap. Phosphine was determined in acetylene produced from two samples of calcium carbide in connection with investigations of explosions of acetylene generators. The remaining 13 samples examined included anthracite ash, mine water, and clay.

Electron Microscope Studies of Coal and of Fischer-Tropsch Catalysts

Although used for other purposes, the electron microscope was applied mainly during the year to studies of coal petrography and catalysis of synthetic liquid-fuel processes. Electron micrographs of aluminum powders revealed particle size and shape differences that accounted for their varying susceptibilities to rapid ignition and explosion. Electron micrographs of carbon blacks resulting from the ignition of acetylene and other gases showed that the particles were spherical and of a range of size similar to commercial carbon blacks.

A large number of electron micrographs has been made in a study of various types of kieselguhrs used as carriers in Fischer-Tropsch cobalt catalysts. Preliminary tests have shown that the type of kieselguhr has some effect upon the catalytic activity. Varieties from Portugal and Germany, presumably used in successful Fischer-Tropsch catalysts (the former in England), and American commercial types have been tested to determine bulk density, pore volume, surface area, chemical analysis, and X-ray diffraction characteristics, and the results have been combined with the electron microscope study in a publication.7/ Figure 1 illustrates the particle size and shape characteristics of some of these kieselguhrs. The magnification of the micrographs, 2,000X, is rather low for the electron microscope, but diatoms are comparatively large specimens for which greater enlargement is not required. The Portugese kieselguhr is characterized by small, compact particles, many of which do not show the porous structure predominating in other types. Disk-shaped structures averaging 5 microns in diameter are the most common regular forms. In the German varieties, such disk-like skeletons are numerous, and nearly all of the particles are of porous regular design. These foreign varieties presumably are from

7/ Anderson, R. B., McCartney, J. T., Hall, W. K., and Hofer, L. J. E., Properties of Kieselguhrs Which may Determine Their Suitability as Carriers in Catalysts: Ind. and Eng. Chem. (In press.)

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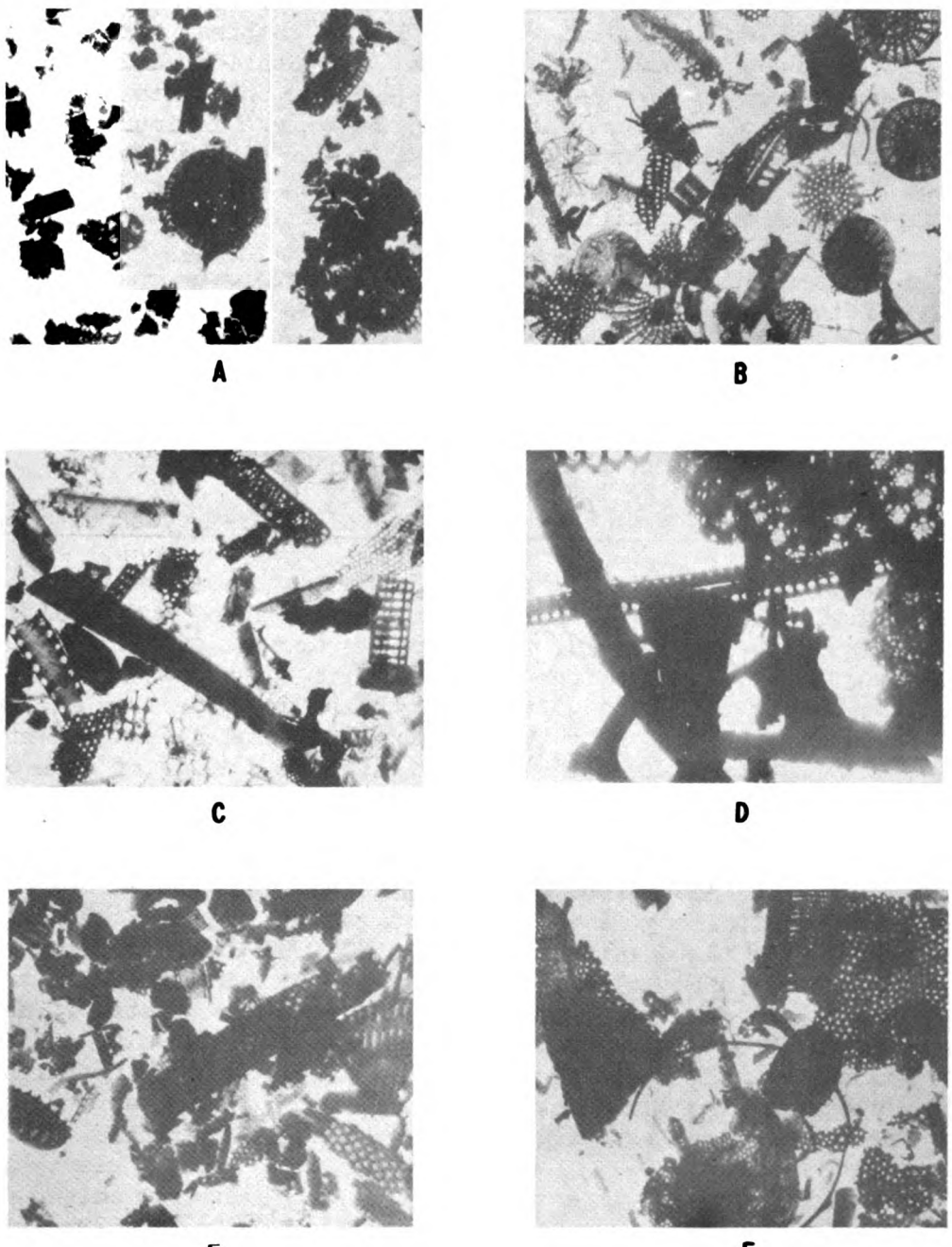

Figure 1. - Electron micrographs of varieties of kieselguhr proposed as carriers in cobalt catalysts for the Fischer-Tropsch synthetic liquid-fuel process. A, Portuguese; B, German; C, Johns-Manville Filter Cel; D, Johns-Manville Hyflo Super Cel; E, Dicalite 911; F, Dicalite 637T. 2,000X.

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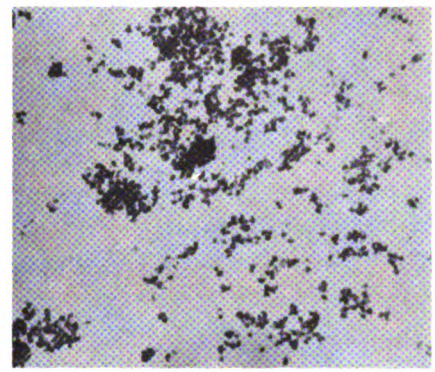
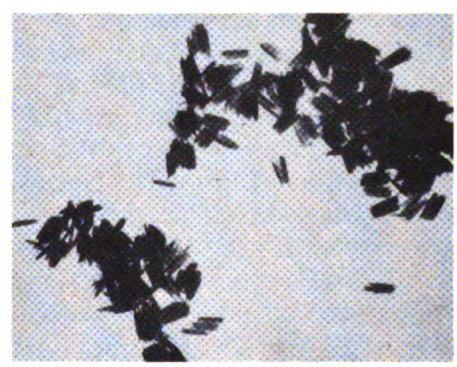


A



B

C

Figure 2. - Electron micrographs of phases of hydrated iron oxide occurring in catalysts for the Fischer-Tropsch synthetic liquid-fuel process. A, α -FeOOH; B, β -FeOOH; C, γ -FeOOH. 5,400X.

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fresh-water deposits. All the American types examined were derived from extensive beds in the Lompoc district of California and are known to be of marine origin. Of a number of grades tested and micrographed, the two shown represent the extremes in particle size. The Filter Cel is a natural material, whereas the Hyflo Super Cel has been flux-calcined. Most of the particles are very porous, with rod and plate forms apparently exceeding the disk type in number. The Dicalite samples come from various American beds, some of fresh-water origin. The two illustrated are from such deposits in Oregon and Nevada. They apparently contain more circular and fan-shaped forms and fewer rodlike particles than the Johns-Manville marine types. Correlation of the physical properties of these kieselguhr carriers with catalytic activity will be attempted in later publications after further testing. This summary of the physical features of various available types is presented for general interest to the users of such materials in industrial applications.

Tests were made on iron catalysts for the Fischer-Tropsch process, prepared in different ways in the search for the basic reasons for their varying degrees of catalytic activity. Electron micrographs have been made of a number of these catalysts and of different forms of iron oxides that occur in them. Some of the latter are illustrated in figure 2, which shows the α, β, and γ forms of the hydrated iron oxide FeOOH. Marked differences in size, shape, and crystal form can be seen. The particles of α-FeOOH are smallest, of fairly uniform size (about 0.1 micron in diameter), and approach spherical shape. The rod-shaped particles of β-FeOOH, about 0.7 micron long and 0.1 micron in diameter, are suprisingly uniform in size. γ-FeOOH also consists of particles of fibrous nature but varying considerably in size. Another common oxide found, α-Fe_2O_3, is similar to α-FeOOH in appearance. These micrographs can be used as standards, in conjunction with X-ray diffraction data, to identify the various phases present in iron oxide catalysts.

A Method of Petrologic Analysis

A new petrologic method has been devised for obtaining comparative information from standard samples of commercial coal. The new procedure combines the high level of optical resolution of componnts inherent in thin-section preparations with a method of sampling that can be expanded to serve any practical accuracy expectation. A new area of petrologic study is opened for investigation by this method to include such diverse materials as deduster dusts, carbonization blends, and fractions of coal from various stages of preparation schedules. The crux of coal sampling has always been to obtain enough coal particles so that all variations in the coal are represented proportionately. Single pieces of coal were needed for individual sections in all previous studies that utilized the property of light transmission. A prohibitive number of sections was therefore required to obtain adequate sampling of a commercial lot of coal, even if the pieces too tiny to section formed a small enough proportion of the whole to be disregarded. Actually, the fines of a commercial sample should never be disregarded, because this fraction, though it may be a small percentage of

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the whole, represents an extreme of component segregation, and a small percentage of fines influences the petrologic composition of the whole sample to a disproportionate extent.

Lithology of the coal - that is, the occurrence and distribution of components - is important in conditioning segregation of the various petrologic entities. Thus, some coals have an even and consistent enough distribution of diverse elements, so that very little segregation can occur in the sizes produced by normal breakage or crushing. Other coals show important tendencies of component segregation in the same range of sizes because of more irregular distribution of the various constituent materials. In order to determine the effects of lithology upon segregation, it is necessary to use petrologic concepts that have compositional and not lithologic connotation. Both vitrain and anthraxylon have certain lithologic as well as compositional connotations in quantitative petrology, inasmuch as fragments of similar optical character, but which occur in fragments smaller than critical and arbitrary sizes, are excluded from them. In recent work at the Bureau of Mines, vitrain is interpreted as a megascopic ingredient, including bands thicker than one-half millimeter. Anthraxylon in a microdetermined constituent that includes all bands thicker than 14 microns. These bands correspond to vitrain, so far as composition is concerned. The complete amount of material in a coal sample that is preserved in the same way as anthraxylon or as vitrain is referred to as vitrinized. This constitutes a larger fraction of coal and one that is not defined by reference to any thickness of occurrence. It is regarded, therefore, as a primary component, and it can be measured at high magnification by the pulverized-coal method to establish the influence of lithology upon petrologic composition.

Coal prepared for commercial utilization is frequently crushed, so that composition rather than lithology is a dominant consideration. Pulverized-coal analyses can be used comparatively to establish what influence petrologic composition has upon utilization properties. Because of limitation in sampling, this phase of petrologic study could not be undertaken systematically previously. It now seems feasible, as a routine method, to determine the petrologic composition of prepared fractions and of other commercial-type samples with a comparative accuracy of about one-half of one percent.

The method involves careful hand crushing of the sample for analysis to pass an 80-mesh screen. The minus 80-mesh sample is then separated into a fine and coarse fraction by means of a 325-mesh screen. The two resultant fractions are compressed into 1-inch-diameter briquettes by using a small amount of water-glass binder and approximately 60,000 p.s.i. pressure. The briquettes are dried and further hardened by impregnating with carnauba wax (m.p. ca 85°C.). Resultant briquettes are hard and can be sawed, smoothed, and polished by the same methods used in sectioning integral coal specimens. Skillful preparation results in sections that show excellent resolution of the components of randomly oriented particles.

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Quantitative determinations of constituents are made at 450X occular magnification. At this magnification, even the most dispersed components are clearly resolved; the largest individual particles in the 80 x 325-mesh briquette are smaller than the single field of view, and so no small group of particles can fortuitously monopolize the quantitative determination.

The primary entities determined include (1) vitrinized matter; (2) yellow, waxy matter (spore coats, cuticles, and other similar optical material); (3) various forms of opaque matter, including fusain; (4) translucent mineral matter; (5) pyritic mineral matter, and (6) open interstitial space. Determinations are prepared on a visually pure basis by arithmetic rejection of items (4), (5), and (6). In other words, significant values are determined as nearly as possible by visual methods on the organic coal substance alone.

In preliminary work, areas equivalent to 100 solid micro fields of visually pure coal substance have been determined for each sample. Areas determined are selected on an arbitrary 1/2-millimeter spacing grid on each section to avoid any possibility of bias due to selection of areas. Each of the pulverized-coal sections is studied proportionately to the percentage of sample represented by their respective briquettes. Thus, if the 80- by 325-mesh briquette equals 80 percent of the sample, determinations equivalent to 80 solid micro fields of visually pure coal are established from it.

Subsequent to proportional study of slides prepared from both coarse and fine fractions of a sample, the results are combined arithmetically to give the final compositional values for the whole of the original sample. Precautions are taken against any form of segregation or differential loss or inequitable determination of compositional constituents throughout the sequence of operations, with the exception of the one sizing operation using the 325-mesh screen. This division of the sample has seemed necessary in order to make the briquettes hard enough to withstand preparation of excellent-quality sections. It is hoped that modification of briquetting procedure may serve later to obviate the need for dividing the samples into two size fractions. The method as now developed is practical in application and has provided information of relative precision on samples not amenable to detailed high-resolution microscopy by any previous method. It further has the distinct advantage of utilizing the same light transmission properties of the coal that have been extensively reported previously in analysis of column specimens of American coal beds. No question, therefore, enters regarding the essential compatibility of the petrologic entities reported by the two methods of analysis.

The following tabulation of analyses (table 1) indicates the nature of samples and results that are being obtained by this pulverized-coal method of analysis.

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TABLE 1. - Petrologic analyses of pulverized coal samples.
(All results expressed on a visually-pure basis.)

	Reactive components		Organic inert components
	Vitrinized matter, percent	Yellow (waxy) matter, percent	Opaque plus fusinized matter, percent
Pittsburgh coal bed - whole experimental sample	92.9	0.6	6.5
Pittsburgh coal bed - air-separated dust fraction (approximately minus 100-mesh)	87.2	.5	13.3
Pittsburgh coal bed - concentrate fraction following elestrostatic preparation of minus 10-mesh coal	96.2	.6	3.2
Herrin (Illinois No. 6) coal bed - crushed raw screenings	94.5	.4	5.1
Herrin (Illinois No. 6) coal bed - air-separated dust fraction (approximately minus 100-mesh) .	71.0	.3	28.7
Herrin (Illinois No. 6) coal bed - concentrate fraction following electrostatic preparation of minus 10-mesh coal	97.6	.6	1.8
Pittsburgh coal bed - 70 percent fraction of 1/4- x 10-mesh coal from experimental head sample ..	93.7	.9	5.4
Pittsburgh coal bed - 30 percent fraction of minus 10-mesh coal from experimental head sample ..	92.0	1.0	7.0
Pittsburgh coal bed - 64 percent float fraction at 1.50 sp.gr. from 1/4- x 10-mesh experimental sample	94.0	1.2	4.8
Pittsburgh coal bed - 17 percent concentrate fraction obtained by experimental electrostatic preparation of 10 x 100-mesh coal ..	96.1	.7	3.2
Herrin (Illinois No. 6) coal bed - 21 percent fraction of tipple sample obtained by experimental electrostatic preparation of 10 x 100-mesh coal	95.0	1.5	3.5

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It is apparent that petrographic composition of different coal fractions varies through a considerable range, even for the Pittsburgh coal bed, which is well-known for its general uniformity. Doubtless there are many coals that show component segregation and concentration due to various causes that are more extreme than those tabulated.

One of the primary objectives of investigations by this method involves preparation of coal with a minimum concentration of organic inert matter for use in experimental hydrogenation. Both opaque matter and fusain are very difficult to liquify, and one of the desirable qualities for an ideal coal for hydrogenation is that it contains a minimum of such inert organic material.

Inert Organic Matter in Eastern Coals

It has been recognized for many years that certain of the organic constituents of coal differ in reactive properties and could, in certain instances, modify the behavior of coal. A concentration of these constituents beyond a certain limit influences the carbonization properties adversely, and these constituents contribute heavily to the unreacted residue in the carbonization process. Unless they are present in an unusually pure concentration, their effect upon combustion reactions is imperceptible, because reactions are influenced most by the most reactive of the materials present. The concentration of more inert organic materials is most desirable where agglomerating properties are deleterious, as in gasification processes or where coal with free-burning characteristics is essential.

In most, if not all, of the eastern American banded coal deposits, the more inert constituents occur in varying concentration through different layers of the coal beds. Some of the nonbanded, cannel, or boghead coals have substantially uniform composition with a high concentration of inert constituents. More significantly, an even greater proportion of these coals lacks agglomerating properties, because spore coats and other waxy components are reactive but do not form a viscous melt on heating. However, the nonbanded coals are local or sporodic in occurrence and usually are associated to some extent with the banded varieties. The important source of weakly caking eastern coal for gasification or allied uses is the beds of banded coal with splinty layers. Splint coal differs in important physical respects from associated bright bands and frequently is amenable to separation and concentration by preparation practices. The splinty bands generally are zones of opaque attritus concentration. Opaque attritus and fusain are inert organic constituents most conducive to weakly caking properties in bituminous rank coal.

As a guide in selecting coals most appropriately considered as potential sources of weakly caking coal, table 2 has been compiled from the many analyses made during the past 15 years. The analyses are arranged in the order of decreasing content of organic inert constituents and range from nearly 50 percent in the case of a cannel deposit in the Elkhorn coal bed to those containing 20 percent inert matter.

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TABLE 2. - Petrologic analyses of coals, with noteworthy concentrations of organic inert materials (fusain and opaque attritus)

Coal bed, mine and mining company, locality (county and State), date of sampling and analysis	Anthraxylon, percent	Translucent attritus, percent	Opaque attritus, percent	Fusain, percent	Organic inert matter, percent	Remarks
Elkhorn coal bed Standard mine, Standard Elkhorn Colliery, Inc. Garrett, Floyd Co., Ky. Sampled 1938; anal. 1938	Nil	51	49	Nil	49	Cannel coal 42 inches thick.
Van Lear coal bed Piedmont mine, Kentucky Block, Cannel Coal Co. (?) Caney, Morgan Co., Ky. Sampled 1938; anal. 1939	Nil	55	45	Nil	45	Cannel-boghead; a 15-inch lump specimen was examined; coal is 32 to 26 inches thick below sandstone roof.
High-Splint coal bed Closplint mine, Clover Splint Coal Co. Closplint, Harlan Co., Ky. Sampled 1930; anal. 1941	25.5	37	30.5	7	37.5	Splinty 7-inch block specimen selected from banded coal.
Cedar Grove coal bed Island Creek mine, Island Creek Coal Co. Holden, Logan Co., W. Va. Sampled 1942; anal. 1942	Nil	64	36	Trace	36	Boghead-cannel 8-inch block specimen from upper part of coal bed
Winefrede (Dorothy) coal bed No. 10 mine, Carbon Fuel Co. Notomine, Kanawha Co., W. Va. Sampled 1936; anal. 1936	17	48	33	2	35	Splinty, banded coal 64¼ inches thick; coal No. 43, carbonization test series.
Elkhorn coal bed No. 204 mine, Consolidation Coal Co. Jenkins, Letcher Co., Ky. Sampled 1930; anal. 1938	17	50	31	2	33	Splinty, 5-inch block specimen selected from banded coal.

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TABLE 2. - Petrologic analyses of coals, with noteworthy concentrations of organic inert materials (fusain and opaque attritus) (Cont'd.)

Coal bed, mine and mining company, locality (county and State), date of sampling and analysis	Anthraxylon, percent	Translucent attritus, percent	Opaque attritus, percent	Fusain percent	Organic inert matter, percent	Remarks
Cedar Grove coal bed Island Creek mine, Island Creek Coal Co. Holden, Logan Co., W. Va. Sampled 1938; anal. 1938	Nil	*70	30	Nil	30	Boghead cannel, 20-inch block specimen from upper part of coal bed.
Upper Cedar Grove coal bed Junior mine, Red Jacket Consolidation Coal & Coke Co. Red Jacket, Mingo Co., W. Va. Sampled 1935; anal. 1935	33	37	28	2	30	Splinty, banded coal 48¼ inches thick; coal No. 39 carbonization test series.
Eagle coal bed Mallory No. 3 mine, Mallory Coal Co. Mallory, Logan Co., W. Va. Sampled 1936; anal. 1936	42	30	26	2	28	Splinty, banded coal 32½ inches thick; coal No. 46, carbonization test series.
High Splint coal bed Closplint mine, Clover Splint Coal Co. Closplint, Harlan Co., Ky. Sampled 1937; anal. 1938	31	41	26	2	28	Splinty, banded coal 40 inches thick; coal No. 54, carbonization test series.
Elkhorn coal bed No. 204 mine, Consolidation Coal Co. Jenkins, Letcher Co., Ky. Sampled 1930; anal. 1930	36.6	35.5	24	3.9	27.9	Splinty, banded coal 90.6 inches thick; coal No. 2, carbonization test series.
Clintwood coal bed No. 1 and 2 mines, Buchanan County Coal Corp. Big Rock, Buchanan Co., Va. Sampled 1933; anal. 1935	41	33	24	2	26	Splinty, banded coal 53.5 inches thick; coal No. 31 carbonization test series.

*Includes 42 percent algae, 14 percent spore coats, and 14 percent humic and resinous matter.

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TABLE 2. - Petrologic analyses of coals, with noteworthy concentrations of organic inert materials (fusain and opaque attritus) (Cont'd.)

Coal bed, mine and mining company, locality (county and State), date of sampling and analysis	Anthraxylon, percent	Translucent attritus, percent	Opaque attritus, percent	Fusain, percent	Organic inert matter, percent	Remarks
Stockton (Lewiston) coal bed						
Sharon mine, Wyatt Coal Co.						
Sharon, Kanawha Co., W. Va.						Splinty, banded coal 46.4 inches thick.
Sampled 1939; anal. 1939	35	39	24	2	26	
Alma coal bed						
Red Jacket No. 6 mine, Red Jacket Coal & Coke Co.						Splinty, banded coal 47½ inches thick; coal No. 36 carbonization test series.
Red Jacket, Mingo Co., W. Va.						
Sampled 1935; anal. 1935	44	31	23	2	25	
Lower Banner coal bed						
Keen Mountain mine, Red Jacket Coal & Coke Co.						Cannel specimen, 4-inch block 39½ inches from floor of bed.
Hanger, Buchanan Co., Va.						
Sampled 1938; anal. 1938	3	72	23	2	25	
Sharon coal bed						
Jackson Iron & Steel Co. mine						Splinty, banded coal 32 inches thick.
Jackson, Jackson Co., Ohio						
Sampled 1937; anal. 1937	55	21	18	6	24	
Lower Cedar Grove coal bed						
Junior mine, Red Jacket Consolidation Coal & Coke Co.						Splinty, banded coal 60¾ inches thick; coal No. 40, carbonization test series.
Red Jacket, Mingo Co., W. Va.						
Sampled 1935; anal. 1935	38	38	22	2	24	
Eagle coal bed						
Prospect opening near No. 7 mine, Carbon Fuel Co.						Splinty, banded coal 31½ inches thick; coal No. 82 carbonization test series.
Carbon, Mingo Co., W. Va.						
Sampled 1943; anal. 1943	37	39	19	5	24	

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TABLE 2. - Petrologic analyses of coals, with noteworthy concentrations of organic inert materials (fusain and opaque attritus) (Cont'd.)

Coal bed, mine and mining company, locality (county and State), date of sampling and analysis	Anthraxylon, percent	Translucent attritus, percent	Opaque attritus, percent	Fusain, percent	Organic inert matter, percent	Remarks
Van Lear (?) (Millers Creek No. 1) coal bed No. 155 mine, Consolidation Coal Co. Van Lear, Johnson Co., Ky. Sampled 1935; anal. 1935	49	27	21	3	24	Splinty, banded coal 48 inches thick; coal No. 33, carbonization test series.
Upper Banner coal bed No. 9 mine, Clinchfield Coal Corp. Clinchco, Dickenson Co., Va. Sampled 1936; anal. 1936	37	40	21	2	23	Splinty, banded coal 51¼ inches thick; coal No. 42 carbonization test series.
Powellton coal bed Coal Mountain mine, Red Jacket Coal Co. Guyan, Wyoming Co., W. Va. Sampled 1943; anal. 1943	44	33	18	5	23	Banded coal 42 inches thick; coal No. 81 carbonization test series.
Taggart coal bed Dunbar mine, Stonega Coal & Coke Co. Dunbar, Wise Co., Va. Sampled 1930; anal. 1930	53	24	20	3	23	Banded coal 52 inches thick; coal No. 3, carbonization test series.
Straight Creek coal bed Hanby mine, Fox Ridge Fuel Co. Hanby, Bell Co., Ky. Sampled 1939; anal. 1939	6	71	23	Nil	23	Cannel coal layer.
Beckley coal bed Winding Gulf No. 1 mine, Smokeless Fuel Co. Winding Gulf, Raleigh Co., W. Va. Sampled 1935; anal. 1936	40	39	15	6	21	Banded coal 54¾ inches thick; coal No. 41, carbonization test series.

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TABLE 2. - Petrologic analyses of coals, with noteworthy concentrations of organic inert materials (fusain and opaque attritus) (Cont'd.)

Coal bed, mine and mining company, locality (county and State), date of sampling and analysis	Anthraxylon, percent	Translucent attritus, percent	Opaque attritus, percent	Fusain, percent	Organic inert matter, percent	Remarks
Lower Banner coal bed Keen Mountain mine, Red Jacket Coal Co. Hanger, Buchanan Co., Va. Sampled 1938; anal. 1938	30	49	19	2	21	Banded coal 54¾ inches thick; coal No. 58, carbonization test series.
Pocahontas No. 3 coal bed Buckeye No. 3 mine, Buckeye Coal & Coke Co. Stephenson, Wyoming Co., W. Va. Sampled 1938; anal. 1938	60	20	11	9	20	Banded coal 42-5/8 inches thick; coal No. 56, carbonization test series.
Alma coal bed No. 4 mine, Spruce River Coal Co. Ramage, Boone Co., W. Va. Sampled 1931; anal. 1931	31	49	14	6	20	Banded coal 66 inches thick; coal No. 16, carbonization test series.
No. 1 Bell ("Smith" or Owen) coal bed Bell No. 1 mine, Midcontinent Coal & Transportation Co. Near Sturgis, Crittenden Co., Ky. Sampled 1939; anal. 1939	54	26	13	7	20	Banded coal 40½ inches thick; coal No. 61, carbonization test series.
Brookville coal bed Conifer No. 1 mine Near Conifer, Jefferson Co., Pa. Sampled 1933; anal. 1934	50	30	16	4	20	Banded coal 48 inches thick; C. K. Graeber sample No. 1.
Brookville coal bed McClellan coal mine (local bank) 3 miles east of Brookville, Jefferson Co., Pa. Sampled 1934; anal. 1934	43	37	17	3	20	Banded coal ca. 24 inches thick; C. K. Graeber sample No. 4 (?)

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COAL MINING

Experimental Mine and Dust Explosions

Demonstrations and Lectures

Following cessation of hostilities, the restrictions against visitors to the Experimental Testing Station at Bruceton, Pa., were lifted. Numerous requests were received from miners, mine officials, and mining schools for educational demonstrations of the ignition of coal-dust clouds by various means that are commonly found in mines, of the dangers arising from the use of black powder, of the greater safety attained through the proper use of permissible explosives, and of recommended safeguards against explosions. Accordingly, seven large-scale mine-explosion demonstrations were held, which were attended by nearly 3,000 persons. In addition, many smaller demonstrations were given for small groups of visitors, among them field men of explosives companies that require them to visit the station as part of their preliminary training, and technical persons from Australia, Brazil, China, France, Great Britain, Holland, Peru, Russia, and this country.

Inflammability and Explosibility of Industrial Dusts and Powders

Continued investigation of the inflammability and explosibility of dusts produced in industrial plants is being carried out. Over 60 dust samples were submitted by industry for evaluation of the explosion hazard that might be encountered in their production and use. A number of the samples were received after actual explosions, flash fires, and fires of spontaneous origin had been caused by the products. These included peanut cracklings, a byproduct from the extraction of peanut oil, dehydrated, ground citrus peels used as cattle feed and fertilizer; soap powders; bituminous coal from a pulverizer; seacoal used as facing for foundry molds; and gilsonite dust from a mine in Utah in which a severe explosion occurred. Tests on several samples of gilsonite indicated that this dust is considerably more explosive than fine bituminous coal dust; the nature of the gilsonite deposit and the methods of mining are such that application of rock dust for preventing the propagation of explosions is most difficult, if not impossible.

Among the other dust samples tested were metal powders, plastics, drugs, insecticides, dehydrated rice, soybean, coffee, sulfur, activated carbon, coal, pitch, dust from exhaust system of an office building, D.D.T., collucotton, tung hulls and kernels, starch, moss, kelp, rockweed, ingredients of explosives, and other products. Nearly all of these were found to present some degree of dust-explosion hazard under proper conditions. For all dusts investigated, recommendations were made concerning safeguards to be taken in the plants in order to reduce the hazard. In testing insecticides containing fine sulfur powder and noninflammable ingredients, it was found that even mixtures with as little as 10 percent sulfur can cause fairly severe explosions. Sulfur powder can be ground and mixed safely in an inert gas atmosphere containing 10 percent or less oxygen. In connection with

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the proposed use of mixtures of powdered coal-tar pitch and aluminum powder for foundry facings in steel plants, a study was made of the explosion hazards of mixtures containing various proportions of stamped and of atomized aluminum powders. This disclosed that mixtures containing atomized aluminum are not more explosive than the pulverized pitch along, but those containing flaked or stamped aluminum are more hazardous.

Research work was conducted on (1) accurate determinations of the lower explosive limits of dust clouds, (2) development of new types of ignition sources for dust clouds exploded in the laboratory bomb, and (3) studies of the effect of dust concentration upon the energy required to ignite dust clouds by static electrical sparks. Two publications were prepared relating to research work on metal-powder explosions.8/

Review of the present state of knowledge of dust-explosion phenomena has indicated that greater progress in prevention of explosions could be made if scientific knowledge were available as to the basic mechanism of the ignition of dust particles, the mechanism of propagation of heat and pressure waves and of flames through the dust cloud, and of the relation of these phenomena to the physical and chemical properties of the dusts.

Release of Dust-Explosion Pressures to Prevent Structural Damage

Few manufacturing structures can withstand the pressures developed by violent dust explosions in a confined space. Rapidly opening explosion-relieving devices or vents in the enclosing walls or roof afford means of dissipating pressure during an explosion. It becomes important to study the design requirements of such relief vents for reducing structural damage. The first phase in this study has been completed and is the subject of two reports.9/ A short motion picture of this work was made. A few of the more important findings of the study are:

1. Aside from the chemical and physical properties of the dust, the violence of a dust explosion in a given enclosure is affected by the method of formation of the cloud, particularly as it influences the uniformity of distribution of the dust particles, by the position and nature of the source of ignition, by the timing of the ignition relative to the formation of the cloud, and by the concentration of the dust in the cloud.

8/ Hartmann, Irving, and Greenwald, H. P., The Explosibility of Metal-Powder Dust Clouds: Min. and Met., vol. 26, 1945, pp. 331-335. Hartmann, Irving, The Explosion and Fire Hazard of Metal Powders: Metals Handbook, American Society of Metals. (In press.)

9/ Hartmann, Irving, and Nagy, John, Effect of Relief Vents on Reduction of Pressures Developed by Dust Explosions: Bureau of Mines Rept. of Investigations 3924, 1946, 22 pp.
Hartmann, Irving, Pressure Release in Dust Explosions: Nat. Fire Protec. Assoc. Quart., vol. 40, pt. 1, July 1946, pp. 47-53.

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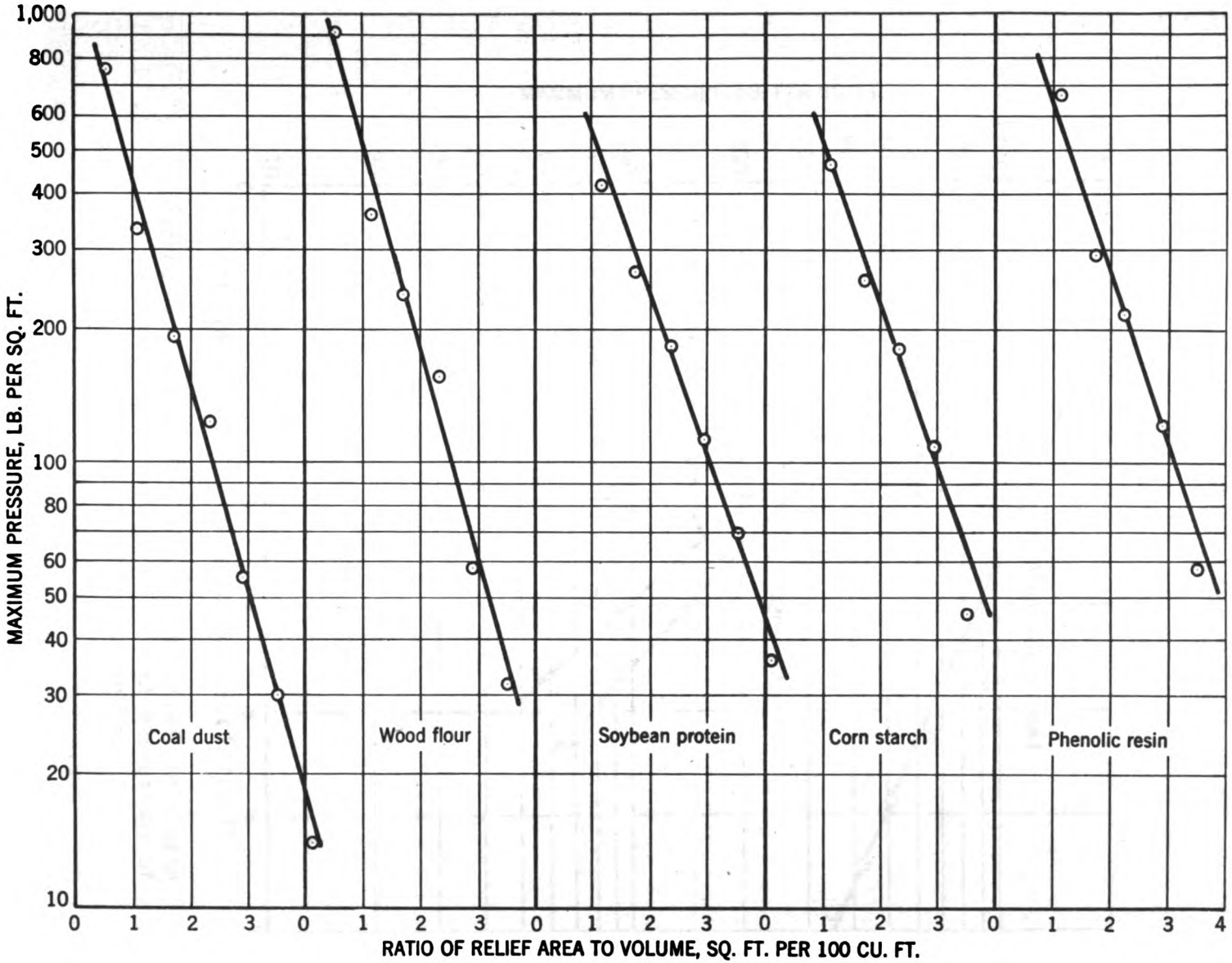

Figure 3. - Effect of unrestricted relief vents upon pressures produced by explosions of various dust clouds.

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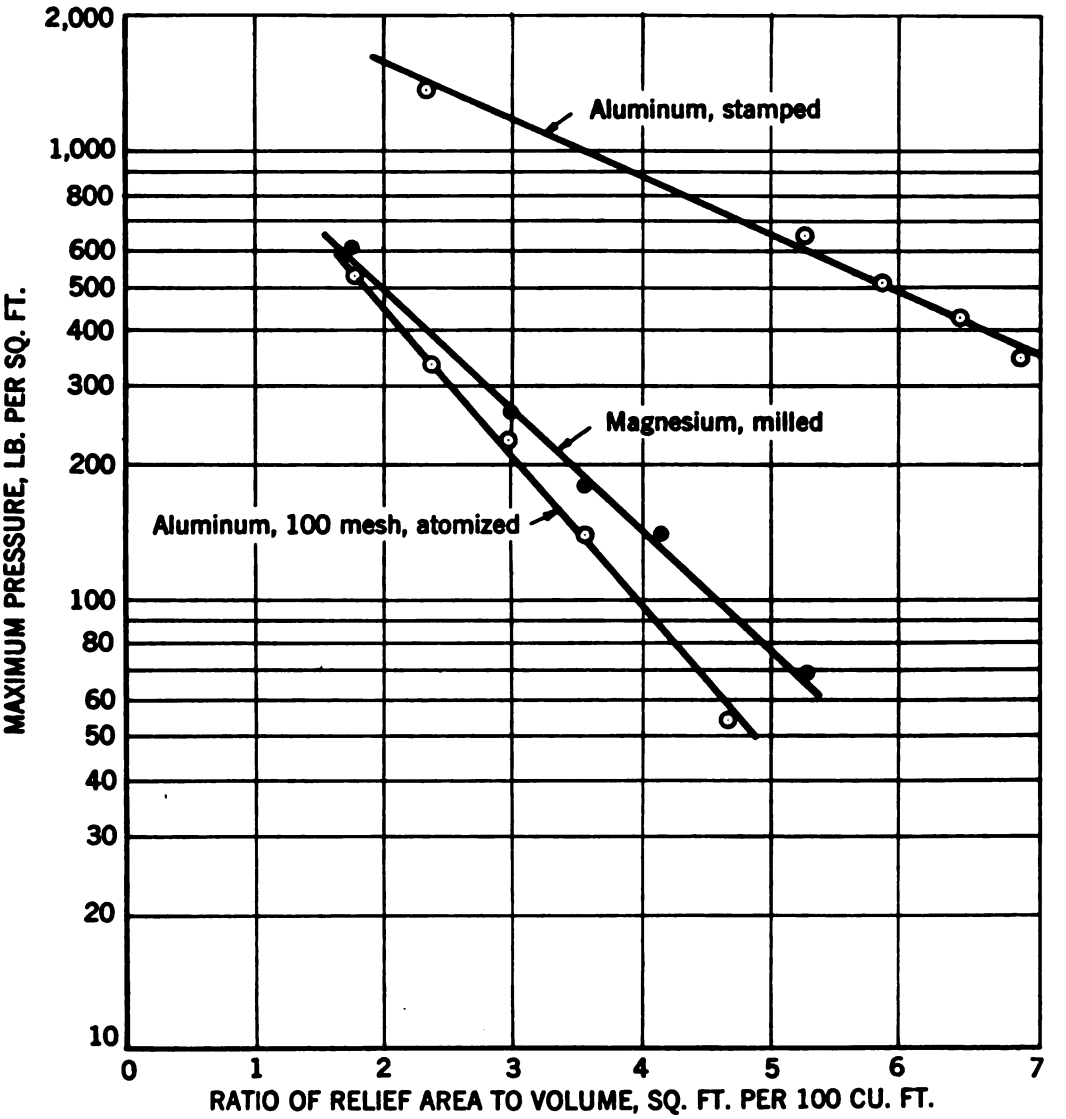

Figure 4. - Effect of unrestricted relief vents upon pressures produced by dust explosions of aluminum and magnesium powders.

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2. Explosions initiated by short electrical sparks are not as violent as those initiated by the flame from a small quantity of guncotton or other ignition source of similar intensity. This is because flame initially raises the temperature of a greater proportion of the dust cloud to the ignition point.

3. Explosions initiated at the instant when all the dust is in suspension in the air produce higher pressures than explosions initiated either prematurely or after some of the dust has settled on the surrounding surfaces.

4. Under similar test conditions, the strongest explosions in the gallery were produced by clouds of stamped-aluminum powder dust. Next in approximately decreasing order of intensity were explosions produced by milled magnesium, atomized aluminum, phenol-formaldehyde resin, cornstarch, soybean protein, wood flour, and coal dust.

5. Data were obtained for all dusts tested, by aid of which the reduction of the maximum explosion pressures with increase in the area of unrestricted or free relief vents could be established. The relations are plotted on a semi-logarithmic scale in figures 3 and 4; they are expressible by an exponential equation of the form:

$$P = Ae^{-kr}$$

where P = maximum explosion pressure
r = ratio of relief vent area to volume of enclosure
A and k = empirical constants, whose values were computed from the test data for each dust

6. In this cubical gallery, explosions could be vented as effectively by several small, unrestricted vents as by a single vent having an area equal to the combined areas of the small vents. This will not necessarily be true of much larger structures or of those of different shapes.

7. To release explosions through vents closed off by heavy-paper or other diaphragms, larger vent areas must be provided than are necessary for unrestricted or free vents.

8. Saw-toothed cutters placed along the peripheries or near the centers of paper diaphragms on vents greatly facilitate their rupture when an explosion starts and permit the use of smaller relief vents than would otherwise be needed.

9. Unrestricted rectangular vents were found to be as effective as square vents of the same areas. Square vents closed by heavy-paper diaphragms proved to be somewhat more effective in releasing explosion pressures than rectangular vents with similar diaphragms.

10. For releasing relatively slow explosions, such as for coal dust, light, hinged, swinging panels are nearly as effective as unrestricted

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vents, but heavy swinging panels are not so effective. For rapid explosions, as with aluminum powder, even vents closed by light swinging panels must have larger areas than unrestricted vents for equal effectiveness.

11. In using swinging panels, windows, or other hinged devices, care must be taken to prevent closure of the relief opening after the initial positive pressure wave of the explosion subsides, in order that destructively high negative pressures should not be developed in the explosion space.

12. The average and maximum rates of pressure rise and the impulses (pressure x time) in the experimental dust explosions decrease with increasing size of relief areas in about the same manner as do the maximum pressures.

Pressure-Relieving Capacities of Glass

In response to request for information on the pressure-relieving capacities of heat-absorbing glass panes to be used in a plant subject to explosion hazards, tests were conducted to determine the explosion pressures at which various types of glass break. The heat-absorbing glass and the double-strength, grade A glass panes were tested unscored, with a light diagonal scoring made with a diamond pencil on the outside surface of the glass (scoring started 2 inches from corners, and there was a 2-inch gap near the center), and also with a deeper diagonal scoring made with a diamond glass cutter. Tests were also made on ribbed glass panes that were not scored. All panes were 1/8 x 14 x 14 inches in size, fixed along all four sides in wood sash set in one wall of the explosion gallery. They were subjected to coal-dust explosions and to more rapid atomized aluminum-powder explosions. A few of the principal test data are given in the following table:

Type of glass	Minimum pressure at which glass broke, lb. per sq. ft.
(a) Coal-dust explosions	
Double-strength "A":	
Unscored	440
Lightly scored	185
Deep scored	70
Heat-absorbing:	
Unscored	260
Lightly scored	90
Deep scored	45
Ribbed glass	440
(b) Aluminum-powder explosions	
Double-strength "A":	
Unscored	250
Lightly scored	110

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Technical Assistance and Service to Others

The Experimental Coal Mine and Dust Explosions Section cooperated with and assisted in the solution of technical problems of other sections of the Bureau, the Department of Agriculture, the War Department, several State agencies, and many national safety groups and industry. These activities included mining, pulverization, and sizing of coals for research in combustion and in synthetic fuels; design of demonstration galleries for dust explosions, and of testing rods for mine roofs; special tests of shale specimens from mine roofs and evaluation of convergence data from mines; furnishing data for design of pressure-release diaphragms and vents for drying ovens, film-processing tanks, dust collectors, and coal-storage bins; advice on explosion prevention in briquetting fine coal, in the manufacture of starch, and in pneumatic conveying of plastics powders; supplying drawings and specifications for construction of apparatus for research on dust explosibility; and preparation of reviews on explosion-pressure manometers and on factors affecting release of pressures in explosions.

Investigation of Coal Deposits

Coosa Coal Field, Alabama

Investigation, by diamond drilling, of the Wattsville Basin in the Coosa Coal Field of Alabama began in December 1945. Diamond drilling in this area indicates that the northern part of the Wattsville Basin may be underlain by a minable area of coal in the Fairview bed. No other coal beds in this area appear to be minable, although future drilling may develop minable reserves in the Broken Arrow and Marion beds in the southern part of the Wattsville Basin.

Analyses of cores of the coal beds in this field show calorific values ranging from 12,000 to 14,300 B.t.u. Agglutinating determinations indicate that the coals are coking. However, in some areas the ash and sulfur content is high.

The investigation was in progress at the end of the year.

Coal Reserves in Areas Where Fuel Supplies are Critical

Toledo Lignite Deposit, Lewis County, Wash.

The lack of sufficient supplies of oil, coal, and wood, the fuels normally used in the Pacific Northwest, caused a critical fuel shortage in that area during the war. Coal was shipped from mines in the Rocky Mountain Region, but the shortage would be relieved by the development of strip mines in this area. The objective of this investigation is to determine the minable reserves in a lignite deposit near Toledo, Lewis County, Wash., which appeared from a reconnaissance to be favorable for strip mining. At the time this investigation was undertaken, the Bureau of Mines had proved a large reserve of high-alumina clay in Cowlitz County, Wash., near the Toledo

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lignite deposit, and the Defense Plant Corporation planned to construct a large plant to process this clay for alumina. A reserve of lignite that could be mined by strip methods would provide a supply of low-cost fuel for such a plant.

The area investigated by diamond drilling comprises sections 15, 16, and 22, T. 11 N., R. 1 E., Lewis County, Wash.

The investigation was conducted under agreements between the Bureau of Mines and the owners, who were private individuals, the State of Washington, and the Northern Pacific Railway Co., which owned the mineral rights in a small tract.

The area is approximately 9 miles east of Toledo, Wash., approximately 8 miles southwest of the Cowlitz, Chehalis & Cascade Railway, and approximately 5 miles north of the Weyerhaeuser Timber Co. railroad, which connects with the Columbia River, a possible rail-water transportation link to industrial centers.

Part of the area has been cleared, and very little timber of commercial value remains on the land (fig. 5). The topography is rolling, and the surface is traversed by deep, narrow ravines, which have been gouged by streams.

Thirty-five diamond-drill holes yielding 2-1/8-inch cores showed that the lignite occurs in a lens comprising two minable benches. The upper and lower benches, separated by a gray-tuff parting about 10 feet thick, average about 25 and 12 feet in thickness, respectively. The reserve of lignite in the lens is estimated at 8,000,000 tons, of which 5,300,000 tons are considered recoverable.

Meta-Anthracite in Newport and Providence Counties, R. I.

The fuel requirements for industrial and domestic consumers in New England, in normal times, are supplied almost entirely by fuel oil, anthracite, and bituminous coal. A shortage of these fuels occurred during the war. Because of this critical fuel situation, an investigation was made of possible sources of fuel nearer to points of consumption in New England.

Meta-anthracite coal, which is a satisfactory fuel for some purposes, had been mined in Massachusetts and Rhode Island many years ago. The coal from mines in the vicinity of Portsmouth on Aquidneck Island, Newport County, R. I., had been used successfully for smelting copper ore and for fuel. The coal is meta-anthracite and does not burn freely. However, the introduction of freer-burning anthracite and bituminous coal into New England markets resulted in abandonment of these mines. With the present-day development of improved equipment for burning coal, methods of burning it probably could be devised, should minable reserves of coal be found.

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Figure 5. - Part of area explored at Toledo, Wash.

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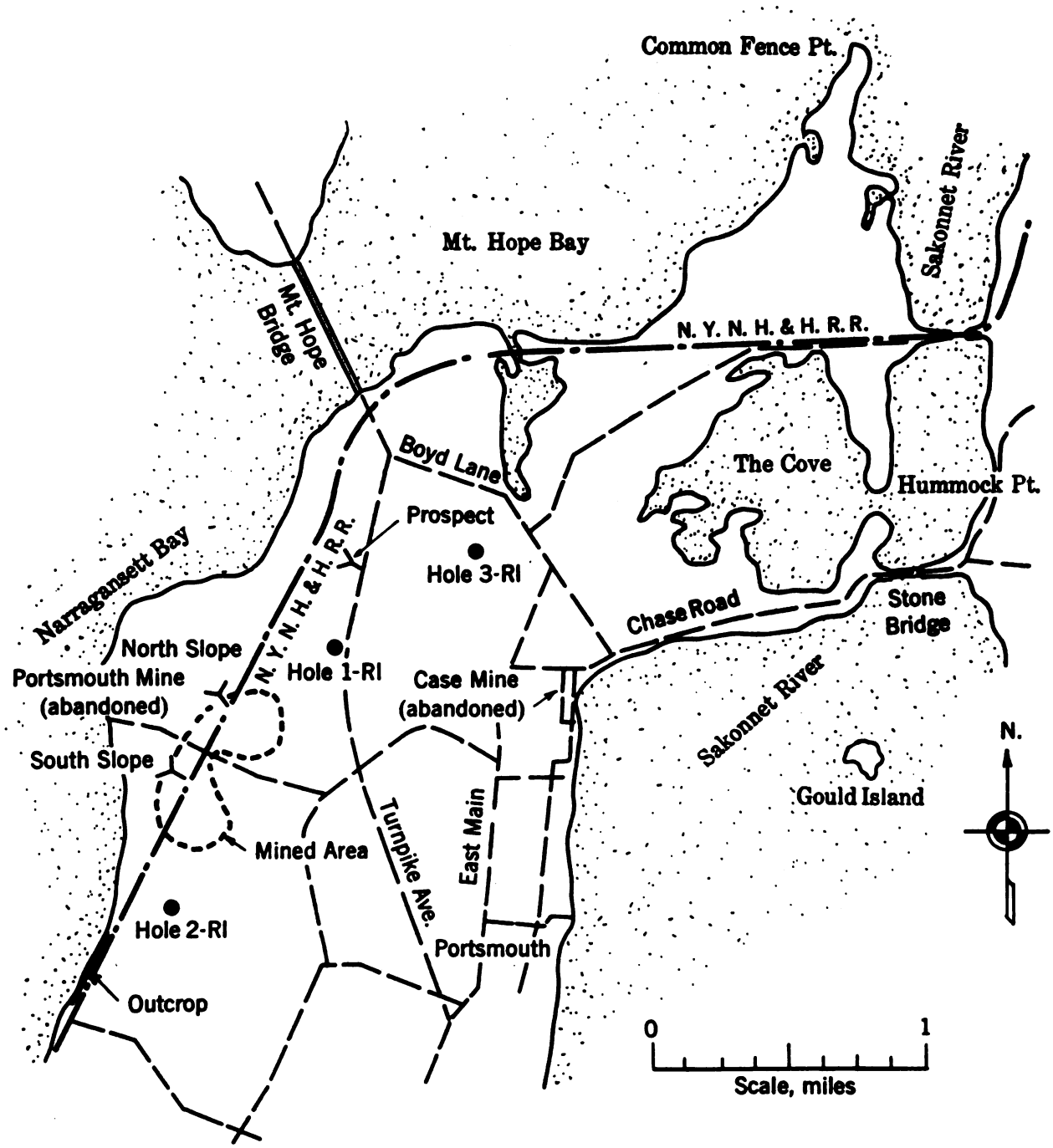

Figure 6. - Diamond-drill hole locations at Portsmouth, Newport County, R. I.

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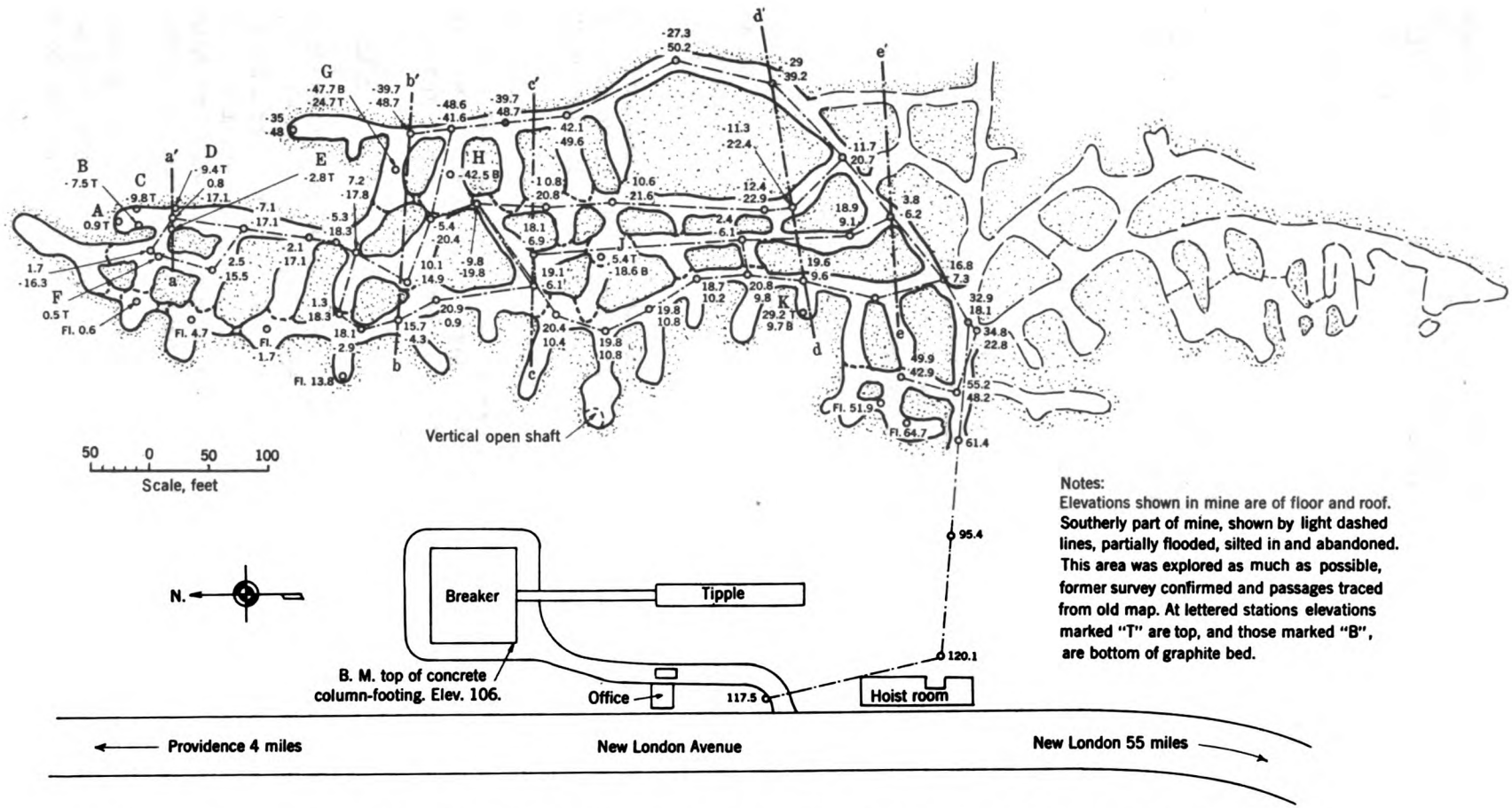
Vertical open shaft
50 0 50 100
Scale, feet
Notes:
Elevations shown in mine are of floor and roof.
Southerly part of mine, shown by light dashed lines, partially flooded, silted in and abandoned. This area was explored as much as possible, former survey confirmed and passages traced from old map. At lettered stations elevations marked "T" are top, and those marked "B", are bottom of graphite bed.
N.
Breaker
Tipple
B. M. top of concrete column-footing. Elev. 106.
Office
Hoist room
Providence 4 miles
New London Avenue
New London 55 miles

Figure 7. - Map of mine, Graphite Mines, Inc., Cranston, R. I. (From survey and map by F. O. Rose, Brown University, Providence, R. I.)

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The purpose of this investigation was to determine sources of additional fuel; and as coal mined at Portsmouth had been used with some success, diamond drilling was undertaken to determine the characteristics and continuity of the beds in this area.

Three vertical diamond-drill holes to recover 2-1/8-inch cores were drilled from surface locations on the north end of Aquidneck Island in the town of Portsmouth. (Refer to fig. 6.)

The Cranston mine, of Graphite Mines, Inc., also was examined. This mine was mapped (refer to fig. 7) for the Bureau of Mines under the direction of Franklin O. Rose, associate professor of engineering of Brown University.

The results of the investigation of the Portsmouth and Cranston areas are summarized as follows:

1. Investigation by diamond drilling and geological examination in the Portsmouth area on Aquidneck Island indicates that minable beds of meta-anthracite are not continuous over appreciable areas, and no estimate can be made of possible reserves in this vicinity.

2. The exposed faces in the workings of the Cranston mine of Graphite Mines, Inc., indicate that there is a recoverable reserve of meta-anthracite adjacent to the workings of this mine. The recoverable reserves cannot be estimated, because the structure and thickness of the bed may change sharply in any direction. The bed may "pinch" out or may be dislocated by faulting.

3. Petrographic examination of cores and mine samples shows that most of the coal of the Narragansett Basin has been subjected to intensive minor faulting and brecciation.

4. The dry ash ranges from 76.1 to 93.7 percent and total carbon from 1.8 to 19.5 percent. With a single exception, the analyses of the cores compared with analyses of meta-anthracite from Cranston and Portsmouth mines showed that the other cores cannot be classified as coal.

Coos Bay, Oregon

The construction of Army camps at Medford and Klamath Falls, Oreg., and the Naval Air Base at North Bend, Oreg., increased the demand for coal far beyond production of the small mines in the Coos Bay area. Coal must be imported from mines in the Rocky Mountain Region to supply any such increased demand and places a burden upon the railroads. The development of a nearby source of fuel would greatly relieve this transportation problem.

The Coos Bay Coal Field, adjacent to the many navigable sloughs extending from Coos Bay, an arm of the Pacific Ocean, and traversed by a branch line of the Southern Pacific Railroad, was a logical source of fuel.

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Fifteen diamond drill holes to recover 2-1/8-inch cores were drilled in the Coos Bay area. Cores of the coal beds penetrated were analyzed at the Central Experiment Station, Pittsburgh, Pa. Based upon the results of this investigation, 1,020 acres of land were proved to be underlain with approximately 10,800,000 tons of coal, of which 8,600,000 tons are considered recoverable. An additional reserve of 6,193,000 tons is indicated and inferred, of which 4,955,000 tons are considered recoverable.

Alaska

Production of coal in Alaska still is less than demand. Greater production is needed for the Army and for industrial and domestic fuel. Investigation by the Bureau of Mines in the Matanuska Field has resulted in increasing reserves adjacent to the workings of the Eska mine, owned and operated by the Alaska Railroad. Diamond drilling in the Eska-mine area was begun by the Mining Branch and completed by the Bituminous Coal Mining Section. Three diamond-drill holes were drilled during the summer of 1945.

Based upon diamond-drilling results, it is indicated that the reserves in the area adjacent to the Eska mine have been increased by an additional 1,600,000 tons of coal in the No. 5, Lower Chapin, Emery (or No. 8), Shaw, and Martin beds. Nine hundred seventy thousand tons of this reserve are considered recoverable through the workings of the Eska mine.

Coaldale, Nev.

Technical Paper 687, titled "Exploration, Composition and Washing, Burning, and Gas-Producer Tests of a Coal Occurring Near Coaldale, Esmeralda County, Nevada," by Albert L. Toenges, Louis A. Turnbull, James M. Schopf, H. F. Yancey, K. A. Johnson, M. R. Geer and L. L. Newman, was published in 1946. (This investigation was discussed in the 1944-45 Annual Report.)

Mining Methods and Practices

Many comparatively small areas of coal suitable for strip mining exist, but these areas probably could not be mined profitably with large-capacity, modern, electrically powered machines because of the high cost-in-place of this equipment and the time and expense required to transport the machines to the mine site. Scrapers and light excavating equipment are mobile, and the investment cost is not great. These units have been used in mining small areas. A study to determine methods in vogue and the economic considerations involved in the use of this equipment was completed.

A Stripping Operation in Illinois

The operations of a coal strip mine in Illinois were studied. The coal bed at this mine ranges from 2 feet to 4 feet, 6 inches in thickness and contains two small partings. The overburden comprises principally soil and glacial drift. In some areas there are lenses of limestone ranging from 6 inches to 4 feet in thickness in the overburden. The ratio of overburden

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to coal, based upon clean coal, ranges from 10.9 to 35.6. The thickness of the coal bed, character of the overburden, and stripping ratios are similar to those in an area in Ohio, where the opening of a strip mine was considered by an operator, who requested a loan from the Smaller War Plants Corporation. A report on the area, including the estimated cost of operations at the Ohio mine, was prepared for the Smaller War Plants Corporation.

Extraction of Pillars

The extraction of pillars in mines developed in steeply dipping beds presents many problems. The methods used at a mine in Washington were studied. The dip of the bed at this mine ranges from 47 to 60 degrees. A section of the bed is as follows:

Interbedded coal and shale	3' 0" to 4' 0"
Shale parting	1' 6" to 3' 0"
Clean coal	5' 0"
Boney coal (left in footwall)	0' 6"

Rooms in a panel are driven approximately 300 feet long on the strike of the bed. A panel usually comprises three rooms driven 8 feet high on 50-foot centers. The full width of bed between foot and hanging walls is extracted on advance in the lower room in the panel. In the two upper rooms, the 5-foot section of clean coal in the bed is extracted on the advance. Coal in these two upper rooms is transported by scrapers to chutes spaced approximately on 50-foot centers, and the coal passes to a shaking conveyor in the lower room. This conveyor transports the coal to the main chute of the panel. Extraction of the full thickness of the bed in the two upper rooms and the pillars between rooms is done on retreat from the panel.

Mine Mechanization

The improvement in technique of mining coal and the relation of this progress to increased extraction and, hence, conservation of coal deposits were reviewed.10/

The technique of coal mining progressed slowly until the advent of mechanized mining. The introduction of the cutting machine was a step forward, but this machine had little effect upon improving the percentage of recovery of coal. Previously, the physical fitness of the loaders and the total output of the miners fixed the output of a mine. The miner was an independent contractor, and supervision over the miner's working place was superficial. The increased cost of labor with the resultant rising cost of production, which necessitated higher selling prices for the product, and the inroads of substitute fuels (oil and gas) presented the problem of increasing the output per individual with the view of reducing the production cost per ton. Since 1923, the amount of coal loaded mechanically underground

10/ Toenges, Albert L., Mechanization in Coal Mining Makes Rapid Progress: Min. and Met., vol. 27, No. 475, July 1946, pp. 393-395.

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has increased from 0.3 percent to 48.9 percent of the total production from underground mines.

The investment necessary to equip a mine for mechanical loading is great, and in order to reduce the fixed charges per ton, multiple-shift operations were instituted, and the working area was planned so that the maximum tonnage can be obtained from a given territory. In some areas this resulted in changes in the mining system that effected a higher percentage of recovery than was possible when hand-loading methods were used. Hence, the introduction of mechanical loading has been a factor in improving the percentage of recovery in some districts.

Mechanization has brought problems to the industry, particularly in reference to production of a product free from foreign impurities. Where hand loading is used, the miner is able to discard the impurities such as partings, boney coal and clay veins, at the time the coal is loaded. The mobile loading machine is not capable of making this separation at the face but loads the broken coal irrespective of the impurities in the bed. This necessitates erection of mechanical cleaning plants at the mines. The output from these plants is not only of higher quality than the hand-cleaned coal but the coal cuttings from the cutting machines, which had been discarded at some mines, are now loaded underground and successfully cleaned mechanically. The recovery per acre at such properties is increased materially.

Where pillars were not extracted, the rapid advance of faces made possible by the use of mechanical equipment resulted in extraction of all the coal in the working places. This was not always possible in some sections because of roof falls. This increased percentage of extraction was due principally to the shorter time the roof was exposed, and caves were reduced to a minimum.

Various types of mechanical loading devices are in use. These types include mobile loading machines (either track- (fig. 8) or crawler-mounted), conveyors (with or without self-loading heads), and scrapers.

Mobile loading machines are best-suited to the mining of level beds of coal or to working on the strike of pitching beds. However, they have had limited use in working places where the pitch was as much as 12-1/4 percent upgrade and 23 percent downgrade.

Conveyors are either the chain or shaking type and are particularly suited to mining thin beds of coal but are used to advantage in mining thick beds on heavy pitches. As conveyors can be operated in a narrow space, they can be used where the roof is not good. In Alabama, the Southwest, parts of Pennsylvania, West Virginia, and other States where there are thin beds of coal, the use of conveyors has made possible the recovery of coal that could not be mined economically by other methods at this time.

Scraper operation is best adapted to places where the roof does not require close timbering at the face. Irregular bottom or floor, however, is disadvantageous to scraper work. This device is also suited to the extraction

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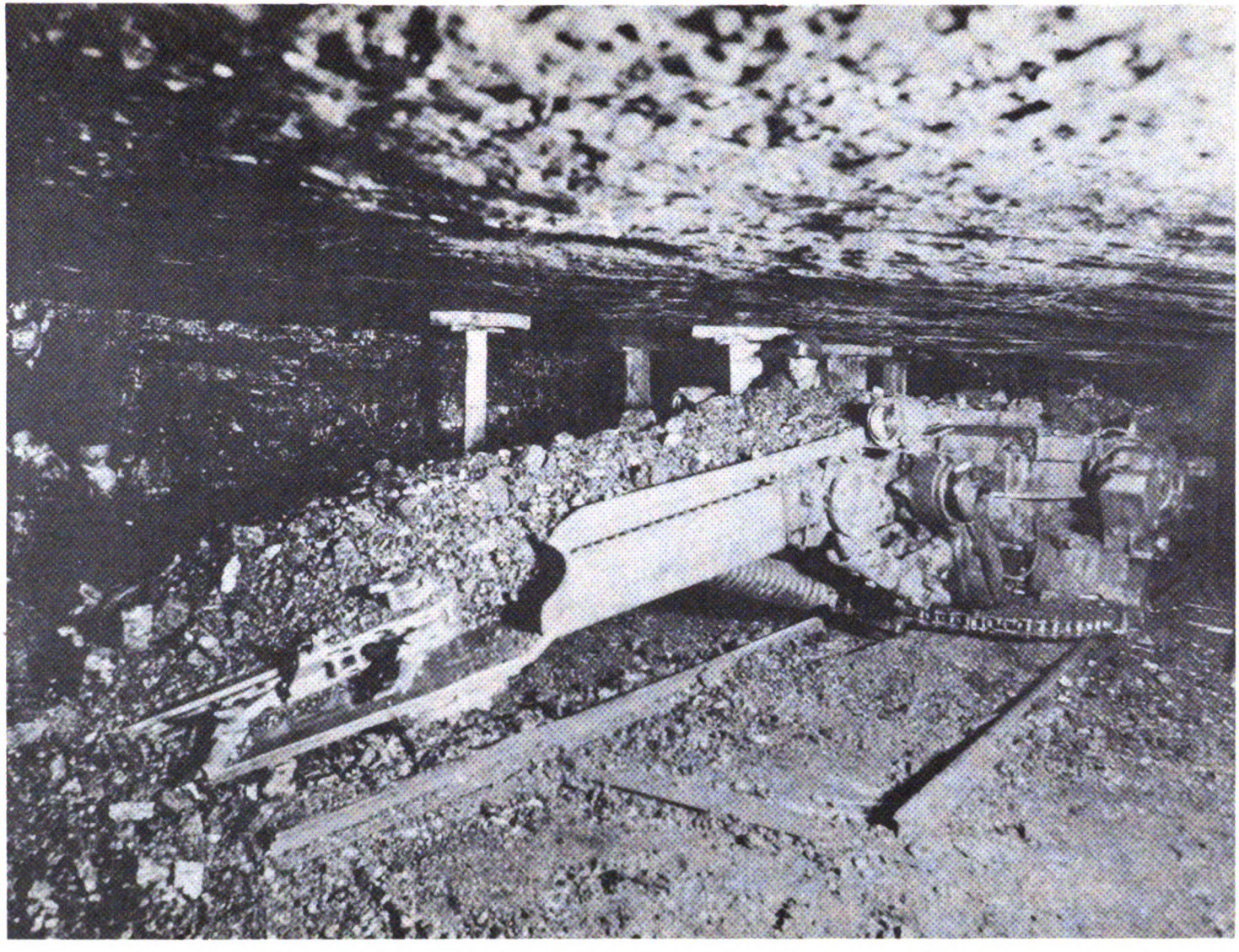

Figure 8. - Mobile coal-loading machine, track-mounted.

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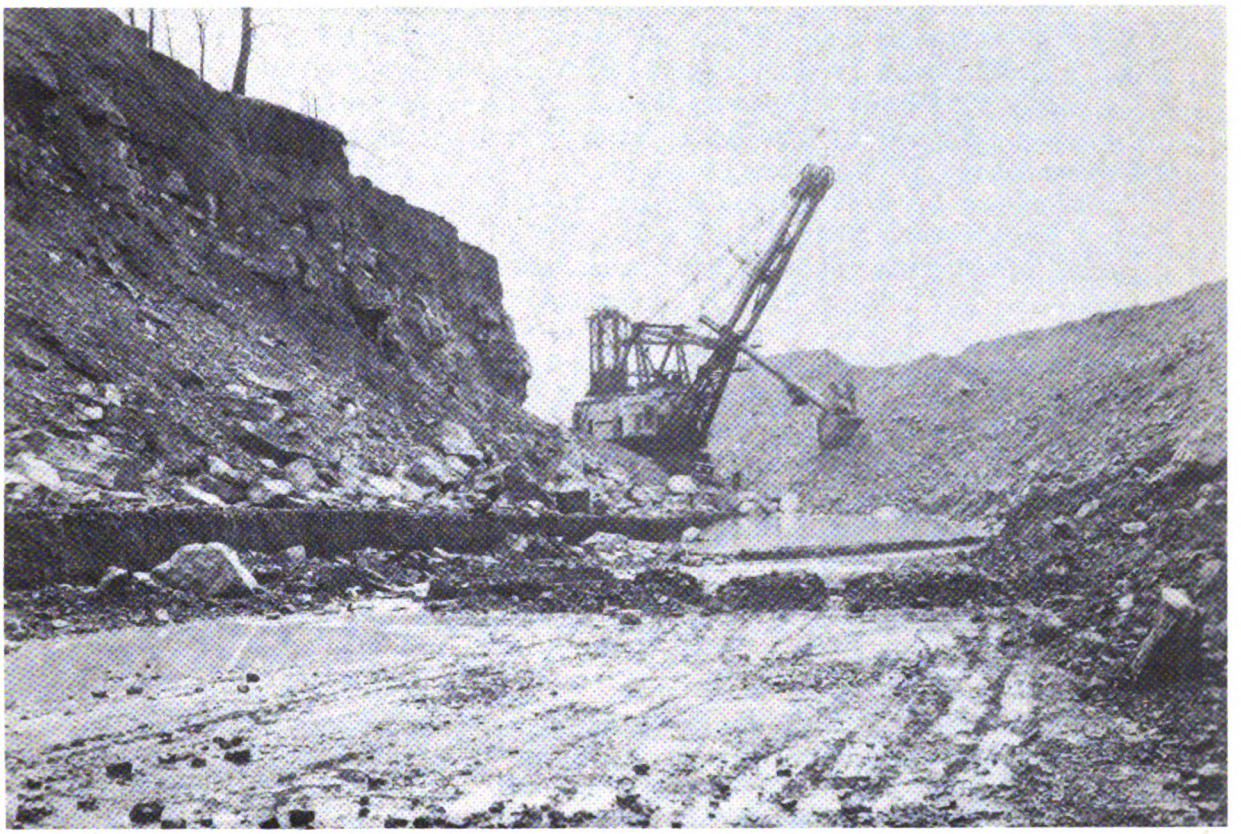

Figure 9. - Electric stripping shovel.

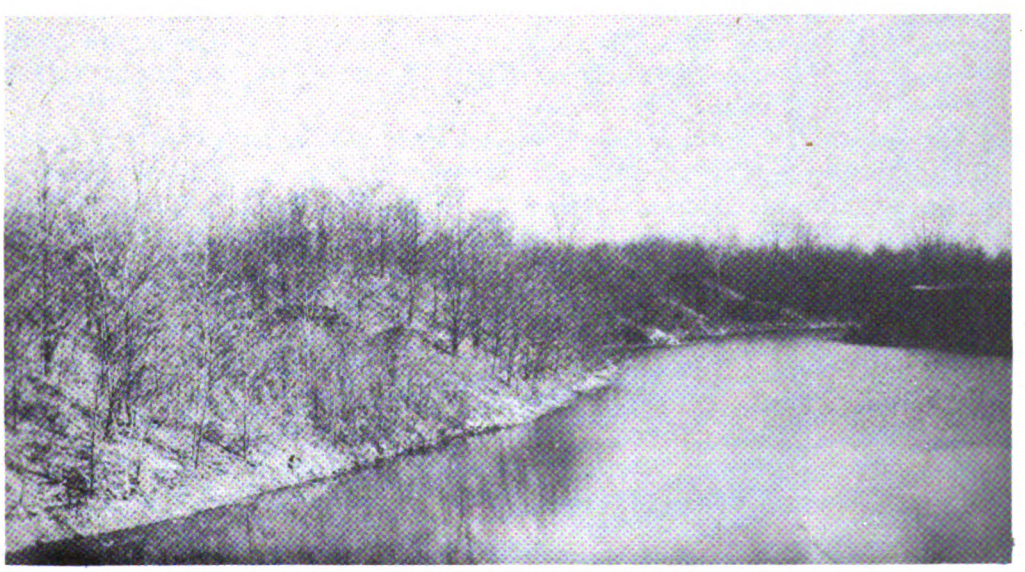

Figure 10. - Planned reforestation of spoil banks.

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of coal from thick, pitching beds where physical conditions do not permit the use of other types of equipment. In order to obtain maximum tonnages per man in a scraper operation, the production from scraper walls and development should be so balanced that a large output per man on walls is not offset by a small production per man from narrow work on development. In thin beds, where scrapers can be used, headings should be driven wide in order to obtain maximum tonnage per foot of entry and to provide space for gob brushing (roof or bottom rock). It would appear that if conditions are suited to scrapers (good roof, hard and smooth bottom with no excessive dips, market for smaller sizes of coal), these will show a low total cost per ton (slightly less labor cost but considerably less investment) than conveyors in coal beds of the same thickness.

As the thicker beds of coal approach depletion, thin beds may be mined economically with mechanized devices for loading the coal. The rapid development of loading equipment, however, has added to the complexity of mechanical mining. Careful planning is required for the return of capital invested in equipment before obsolescence occurs. Coordination of every operation is necessary. A study of methods coordinating transportation with loading is essential and should show the way to increase the output per unit and, hence, reduce the cost per ton. The men working with the machines should be properly trained and educated to conduct the operations safely.

In recent years there has been rapid progress in the technique of mining coal by the stripping method, which is considered mechanical mining. Where the topography of the area and the depth to the coal bed are favorable for stripping, a large percentage of the total production of coal is obtained by this method. The production of strip coal has increased rapidly in some States, and for the United States it has increased from 2.8 percent in 1924 to 13.5 percent in 1943.

In 1943, strip mines in Alabama produced 427,039 tons, which was 2.5 percent of the total output of coal mined in the State.

Technique in strip mining has advanced from the use of teams and scrapers in the early days to electric-powered shovels of large capacity (35 cubic yards) of today. (Refer to fig. 9.) Electric coal-loading equipment has been instituted to replace hand loading. Both rail and motorized equipment are used today to transport the coal from the mines to the tipple, and the coal is prepared mechanically.

Strip mining is particularly adapted to areas where the coal bed outcrops or lies at comparatively shallow depths - 60 feet or less (at greater depths for short distances). The thickness, physical and chemical characteristics, and dip of the coal bed, the composition of the overburden, and the topography govern the extent of mining in strip areas.

From the layman's point of view, strip mining appears a simple procedure. However, this method of mining has many pitfalls, and unless a thorough

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and extensive study is made of the many factors involved, the venture is bound to fail. The investment in equipment is great; therefore, the proven reserves must be large to reduce the fixed charges. Exploration should be thorough, especially on beds known to be irregular in thickness and extent. The character of the overburden should be studied to determine the proper method of breaking this material. The many other phases of this method, including drilling, stripping, loading, transportation, and preparation must be given consideration.

The advance made in strip mining have made this method of mining a decided factor in conservation of coal deposits.

The recovery of coal in underground mining in the States where strip mining is important ranges from approximately 50 to 60 percent. Strip mining recovers the maximum amount of coal from a given area, approximately 75 to 95 percent, and is a means of preventing waste of a natural resource that cannot be replaced.

A contraphase of conservation in strip mining is the overturning of the land, which makes it useless for a time. Erosion, weathering, and natural vegetation usually will make lands useful for grazing, recreational areas, forest reserves, and, in some places, agriculture. Years are required to reclaim land by these means, and it has been proved that planned reforestation is a profitable investment.

Experiments in systematic tree planting in Indiana and Illinois showed that this method of reclaiming spoil banks is successful. (Refer to fig. 10.) Operators in other States have followed the example set by operators in these States. Not only can the reclaimed surface be converted to timber lands, but these lands have been used for grazing and fruit orchards. New industries should follow strip mining in these areas where reclamation has been instituted.

Strip mining (which is mechanized mining) and the application of mechanical loading devices in underground coal mines have been factors in improving technique in extraction of coal and in improving recovery, which results in conservation of coal deposits.

Australian Coal Mining

One of the Bureau's mining engineers with recent and extensive experience in Australia described the coal-mining industry of that country in the technical press.11/

He pointed out that Australian coal mining has drawn heavily on the American industry for both mining practice and machinery. The mines are highly mechanized, though results are not equivalent to those in this country.

11/ Hawkins, R. A., Australian Coal Mining: Min. and Met., August 1945, vol. 26, No. 464, pp. 382-4.

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Demands for coal are primarily industrial, freeing the industry of much of its dependence upon climatic and seasonal fluctuations.

The States of Tasmania and Victoria generate hydroelectric power; the remainder of the country must depend on an annual supply of 10 to 13 million long tons of bituminous coal, 5 million long tons of brown coal, and on charcoal, wood, and imported fuel oil.

The brown coal comes from a State-operated open pit at Yallourn, Victoria. Approximately 75 percent of the output is consumed by the Victorian Government power stations, and the other 25 percent is briquetted by the State and sold to the general public.

The coal deposits are widely distributed, but most of the tonnage to supply a continent-wide market comes from three districts near Sydney, New South Wales. A large proportion of the production possesses excellent coking properties. Generally, the seams are horizontal, 6 to 14 feet thick, and have frequent local undulations.

The universal system of mining is known as "bord-and-pillar," which is similar to the American block system.

Surface handling consists only of sizing and loading, and sometimes hand picking. In 1945, one company started to construct a heavy-media cleaning plant.

High-grade coal reserves are large, both in pillars and in undeveloped known areas, and could be increased further by the introduction of modern coal-cleaning methods for the lower-grade coals.

Eickhoff Shearing Machine

In introducing and developing new types of equipment and methods for underground anthracite mines to improve the competitive position of anthracite by lowering production costs and to enhance the safety of the mine workers through improved working conditions that will accompany underground modernization, advantage was taken of an opportunity offered by the Technical Industrial Intelligence Committee, G-2 Division, SHAEF, for Bureau of Mines personnel to visit British and German mines and plants of mine-equipment manufacturers. Two lightweight German shearing machines, which are unlike chain cutting machines manufactured in the United States, were received, completely overhauled, and are now ready for underground testing. These machines are thought to be especially suitable for winning coal in thin, steeply pitching beds as soon as gangway development into a separate working section has been completed.

The Coal Planer

The rigid-blade German coal planer, which was uncovered by the Bureau's investigators as the principal European wartime development in underground

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mining equipment,[12] operated along a face 750 feet long and mined 900 tons per shift, is not suitable for planing anthracite without alteration. However, design, manufacture, and development of a new type of vibrating-blade coal planer have been arranged for by the Bureau of Mines. The selection of a location for experimental longwall mining is under consideration. Designed for longwall work only, in beds of moderate pitch, the possibilities of its basic principle of simultaneous mechanical winning and loading without cutting or blasting can be determined only after it is proved that anthracite can be planed and an opportunity is given engineers to study the coal planer in actual operation. Its successful development would open the way for attainment of the continuous mining principle and would eliminate frequent unproductive shifts.

Scraper-Slide and Shaker-Conveyor Loading Machine

Delay in delivery of steel parts and stock for the manufacture of the Bureau of Mines combination scraper-slide and shaker-conveyor loading machine for use in developing gangways in thin, steeply pitching beds made it necessary to postpone underground tests in the 1946 fiscal year. This loading machine is designed to eliminate transportation delays at the working face by providing for multiple car loading and in this way make possible one complete cycle of drilling, blasting, timbering, and loading each shift for twice the present rate of advance. Final development of this equipment should make possible the providing of more working places by the additional territory opened each year.

Effect of Scrubbing upon the Odorous and Irritating Constituents of Diesel Exhaust Gas

In field studies of the use of Diesel locomotives underground, it has been observed that the odor of Diesel exhaust gas can be detected in the underground atmosphere even when the ventilation supplied is sufficient to dilute each harmful constituent of the exhaust to a concentration considerably less than its permissible maximum. Because the odor of Diesel exhaust gas is objectionable to some individuals, it appeared desirable to obtain information on the feasibility of minimizing or removing the odorous constituents by scrubbing with water or water solutions containing suitable chemicals. Full-scale tests have been made to determine the effect of scrubbing Diesel exhaust gas with water. Bench-scale laboratory tests have been made to determine the effectiveness of solutions of sodium sulfite in water and to study methods for inhibiting the oxidation of sodium sulfite solutions.

From information available at present, it is not possible to state definitely which compounds or constituents of Diesel exhaust gas are responsible for the odor. Aldehydes are present in the exhaust and are known to have odorous and irritating properties. However, other constituents of the exhaust gas, such as unburned or partly oxidized fuel, which are present in

12/ Buch, John W., Mining Thin, Steeply Pitching Seams with the Coal Planer in the German Ruhr District: Mechanization, November 1945, pp. 92-97.

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low concentrations, may also contribute to the odorous and irritating properties of the exhaust gas. Until more information is available, it is necessary to relate the odor and irritation of Diesel exhaust gas to the concentration of aldehydes in the exhaust gas. This is at best an approximation, not only for the reasons outlined above, but also because of analytical difficulties in detecting and differentiating between the different aldehydes that may be present. In the tests described here, total aldehyde concentration was determined by a modification of a method[13] based upon the reduction of silver nitrate by aldehydes, and the result is expressed as equivalent formaldehyde.

Although the limitations of water as a scrubbing medium for Diesel exhaust gas could have been predicted from theoretical considerations, it was considered advisable to obtain experimental data on these limitations to answer numerous inquiries on this subject. Furthermore, the data are of interest in connection with the development of permissible Diesel locomotives, as these will be equipped with water scrubbers for cooling the exhaust gases and quenching sparks.

In the studies described, the exhaust gases from a four-stroke-cycle, four-cylinder, commercial Diesel engine (engine B) were bubbled through a scrubbing tank containing approximately 10 gallons of water for a period of approximately 6 hours. Arrangements were made to replace, continuously, the water lost by saturation of the exhaust gas. Samples of gas taken before and after the scrubber were analyzed for carbon dioxide, carbon monoxide, oxides of nitrogen, and aldehydes. Observations of odor intensity and nasal and eye irritation were made on both the raw and scrubbed gases.

The effect of water scrubbing upon the removal of aldehydes from Diesel exhaust gas is shown in table 3. The results indicate that a portion of the aldehydes present may be removed by water scrubbing. The fraction removed depends on the concentration of aldehydes in the inlet gas, the concentration of aldehydes dissolved in the water, the temperature of the scrubbing water, the surface of scrubbing water exposed, and the time of contact of the exhaust gas in the scrubber. In full-scale tests it is not possible to control many of these variables, and therefore direct comparisons are difficult to make. However, the results of tests at 1,400 r.p.m. indicate that at higher scrubbing temperatures (greater than approximately 134°F.) the removal of aldehydes is negligible when the inlet concentration is of the order of 20 p.p.m. The effect of reduced time of contact is also indicated in comparisons of results of tests at 700 and 1,400 r.p.m. For example, at comparable scrubbing-water temperatures, the percentage removal of aldehydes is considerably less in the tests at the lower time of contact (higher engine speed). Scrubbing the exhaust gas with water removed no carbon monoxide and only a very small fraction of the carbon dioxide. The removal of oxides of nitrogen was negligible.

13/ American Gas Association Research Bull. 15, 1942.

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TABLE 3. - Results of tests on the scrubbing of Diesel exhaust gas with water

Test No.	Engine speed r.p.m.	Engine load, b.h.p.	Volume of dry exhaust gas, cu.ft. per hr.1/	Temperature of scrubbing water, °F.	Concentration of aldehydes, p.p.m. by vol.2/ Inlet3/	Outlet3/	Aldehydes removed; percent of aldehydes in inlet gas
11	596	0.2	2,100	80	38.3	10.4	73
16	684	3.6	2,130	99	10.6	5.7	46
12	699	9.7	2,150	115	27.9	5.8	79
13	706	9.8	2,060	117	14.0	4.0	71
14	1,467	.4	4,400	98	101.6	65.2	36
17	1,429	8.9	4,200	122	51.0	31.8	39
15	1,386	19.2	3,870	134	23.7	22.9	3

1/ Dry gas at 29.92 mm. of mercury and 60°F.
2/ Expressed as equivalent formaldehyde.
3/ Weighted average for 6-hour test.

The results of observations of odor intensity, nasal irritation and eye irritation of the raw and scrubbed gas are shown in figure 11 as a function of concentration of aldehydes in the exhaust gas. The average intensity of each stimulus represents an average of observations of at least five individuals. The intensity of odor or irritation is arbitrarily defined as follows:

Odor intensity	Eye and nasal irritation
0 - No odor.	0 - No irritation.
1 - Very faint; minimum odor, but positively perceptible.	1 - Faint, just perceptible, threshold, not painful.
2 - Faint, weak odor, readily perceptible.	2 - Moderate.
3 - Easily noticeable, moderate intensity.	3 - Strong, discomforting, painful, but may be endured.
4 - Strong, cogent, forcible odor.	4 - Intolerable, cannot be endured.
5 - Very strong, intense effect, may bite or irritate.	

Figure 11 indicates that both odor and irritation are related to the concentration of aldehydes in the exhaust. In every instance a slight reduction in intensity of odor or irritation was observed when aldehydes were removed by water scrubbing. Figure 11 also indicates that the threshold of odor (as indicated by aldehyde concentration) occurs at a concentration of the order of 0.3 p.p.m. and that the threshold of nasal and eye irritation occurs at a concentration of 1 to 2 p.p.m. If we assume that an atmosphere containing 0.3 p.p.m. of aldehydes would be odorless, then exhaust gas containing 30 p.p.m.

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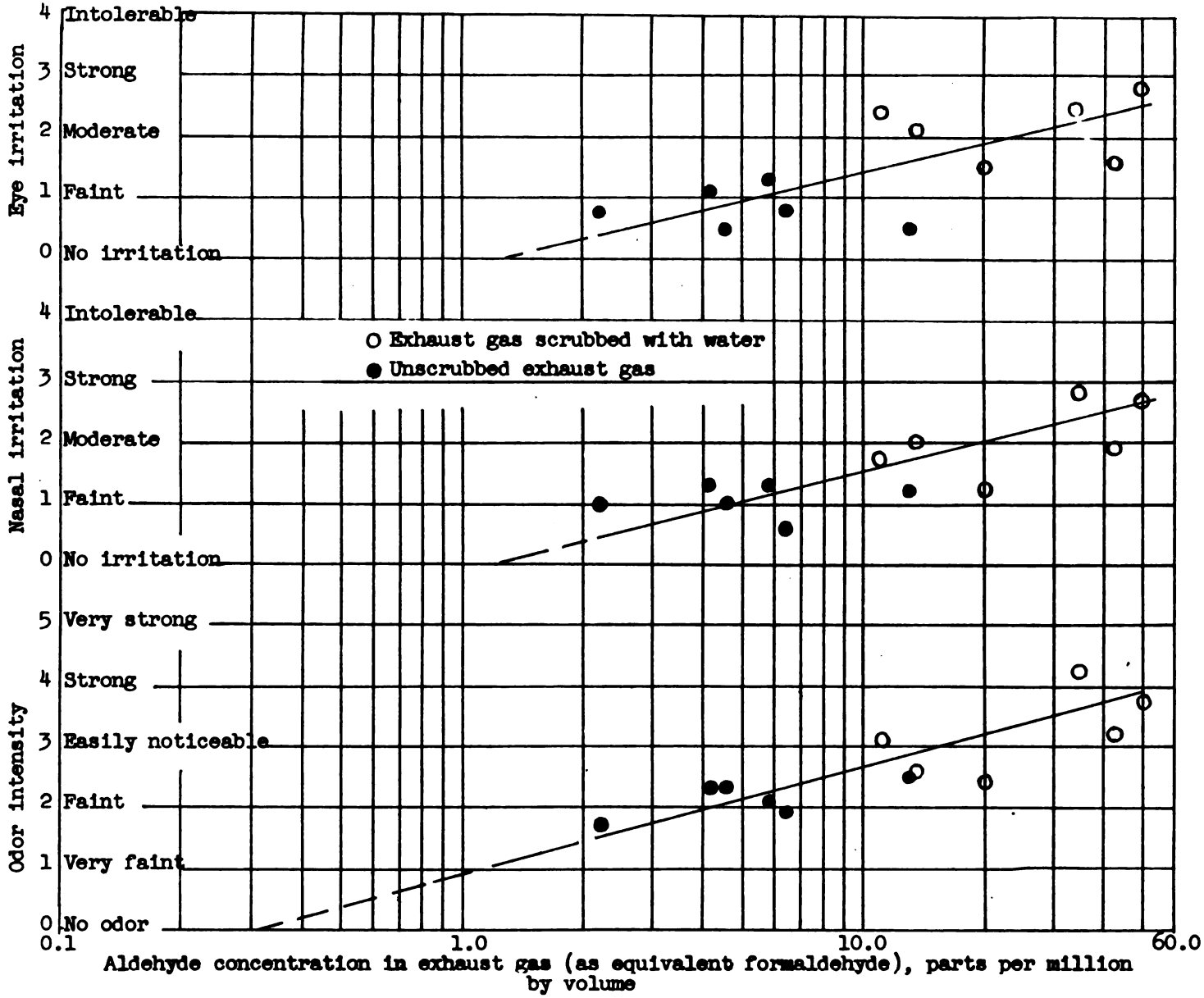

Figure 11. - Relationship between aldehyde concentration and odor intensity and nasal and eye irritation of Diesel exhaust gas.

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of aldehydes would require 100 volumes of air per pound of exhaust gas to produce an odorless underground atmosphere. This dilution is considerably greater than that generally necessary for carbon monoxide and oxides of nitrogen. It is not surprising, therefore, that the odor of Diesel exhaust gas can be detected even when ventilation is adequate for the harmful constituents of this exhaust.

From the foregoing, it is evident that water is not a satisfactory scrubbing medium for Diesel exhaust gas, as under some conditions none of the aldehydes are removed, and even under the most favorable conditions the removal was not greater than approximately 80 percent. It appears desirable, therefore, to seek a more efficient means for removing aldehydes.

Studies are in progress on the removal of aldehydes by solutions containing sodium sulfite. Preliminary laboratory tests have shown that sodium sulfite solutions are effective at temperatures as high as 160°F. However, it is necessary to retard the oxidation of sodium sulfite by the addition of an inhibitor. Suitable inhibitors have been found, and full-scale tests are in progress.

Performance of Diesel Engines

In studies of the effect of barometric pressure upon the composition of the exhaust gases produced by Diesel engines, considerable data were obtained incidentally on engine performance. Analysis of these data led to the development of methods for converting measurements of the power output of Diesel engines to standard atmospheric conditions; a problem that has confronted the Diesel-engine industry for many years. The fundamental theory underlying this problem was developed in 1941,[14] and the recent tests of three different, normally aspirated, commercial, four-stroke-cycle Diesel engines at different barometric pressures permitted an extension and experimental verification of this theory.[15]

Mine Rescue Telephones

The Bureau has experimented with battery-powered telephonic equipment since 1925 in attempts to develop a safe and dependable method of communicating with rescue crews working in dangerous explosive gases. A system consisting of transmitters, receivers, and 1,000 feet of two-conductor cable has been devised and used successfully in actual rescue and recovery work underground. The assembly consists of two dynamic-type receivers, one worn by a member of the crew and one by an attendant at the fresh-air base, one vibration-powered transmitter, one voice-powered transmitter, 1,200 feet of

14/ Elliott, Martin A., A Rational Basis for Correlating Data on Compression-Ignition Engine Performance Obtained at Different Intake and Exhaust Conditions: S.A.E. Jour., vol. 40, 1941, pp. 532-543.

15/ Elliott, Martin A., Conversion of Measurements of Power Output of Diesel Engines to Standard Atmospheric Conditions: Trans., A.S.M.E., vol. 68, 1946, pp. 525-539.

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two-conductor cable, and a reel. The cable is used in place of the rope formerly employed for a signal and life line. The conventional sash-cord type of life line was never satisfactory; frequent failures occurred in transmitting pull or jerk signals owing to fouling of the rope or its elasticity. The greater ease and scope of telephone-voice communication removes much of the difficulty and uncertainty formerly experienced and allows the work to be done more safely and efficiently.16/

Allaying Mine Dust

Coal dust presents an ever-present coal-mine explosion hazard, and persons forced to breath large quantities of it may be made subject to respiratory infection. Dust-allaying equipment for applying water or wetting solutions on the cutter bars of mining machines, on mobile loaders, on belts or conveyors, and on loaded cars is being installed in more mines every year. These installations are far from being standardized, and, to assist in the adoption of the best-known present practices, the Bureau has surveyed available equipment and the installations and systems now in use.17/ A report has been published giving in detail suggested arrangements of dust-allaying equipment as an aid to both manufacturers and mine officials toward obtaining the most efficient systems.

Hazards of Trolley-Locomotive Haulage

Trolley-locomotive haulage has been an important factor in the superior efficiency of production in the underground coal mines of the United States. It has proved to be rapid, economical, and efficient; nevertheless, so many hazardous features and unsafe conditions are involved as to justify the view that the system should be scrapped unless operation can be modified to bring about safe conditions. The hazards have been recognized for years, and although efforts have been made to combat them, the size of equipment and extent of mechanization has increased without finding workable means of safeguarding lives and property against dangers of shock, gas ignitions, and fires. Alternative methods of transportation are being investigated as a solution to the problem in the event that trolley systems cannot be properly safeguarded.18/ Among the methods under study by engineers of the Health and Safety Branch are storage-battery locomotives, conveyor systems, and Diesel locomotives.

16/ Forbes, J. J., Griffith, F. E., Cash, F. E., and Petersen, Max S., Mine Rescue Life-Line Telephone Assemblies: Bureau of Mines Rept. of Investigations 3875, 1946, 9 pp.

17/ Owings, C. W., Suggested Methods for Installing Dust-Allaying Equipment in Bituminous-Coal Mines: Bureau of Mines Rept. of Investigations 3843, 1945, 31 pp.

18/ Harrington, D., and Warncke, R. G., Hazards of the Trolley-Locomotive Haulage Systems: Bureau of Mines Inf. Circ. 7328, 1945, 37 pp.

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Diesel Locomotive Tests

Technicians and engineers of the Health and Safety Branch have investigated the use of Diesel locomotives in mines and tunnels to discover their limitations or advantages. One series of tests, on which a report has been published,19/ was conducted by the Bureau of Mines on the Cascade Tunnel of the Great Northern Railway to determine the effects upon the tunnel atmosphere of a Diesel freight engine. The tunnel was not provided with mechanical ventilation. The observed concentrations of carbon dioxide were not hazardous, nor was the oxygen content of the tunnel air depleted to a significant degree. Carbon monoxide was only slightly about 0.01 percent at any time. Oxides of nitrogen were found in a considerable area behind the train in concentrations exceeding 25 parts per million, usually considered the maximum permissible in working places; persons at work in the tunnel might be affected, but crews on moving trains would not be endangered. The foregoing contaminations were observed for trains pulling upgrade; no appreciable contaminations were observed for downgrade trips. Smoke from the locomotive reduced visibility in the tunnel.

Liquefied Natural-Gas Fire in Ohio

On October 20, 1944, a disastrous fire occurred at the liquefaction, storage, and regasification plant of the East Ohio Gas Co., Cleveland, Ohio,20/ as the result of failure of an insulated cylindrical tank in which liquefied natural gas was stored at less than 5 pounds per square inch pressure and at a temperature of minus 250°F. Eye witnesses of the failure of the tank reported that streams of liquid or fog issued from its side, and that almost immediately the tank opened and discharged its entire contents over the plant and adjoining property at a lower elevation. The gas entered storm sewers, was ignited, and exploded. Heavy loss of life and property resulted. The Bureau investigated the disaster to obtain technical information that might help to prevent similar occurrences; representatives of the Health and Safety Branch joined with members of the Fuels and Explosives Branch in the investigation and in the report issued in 1946. Definite conclusions were not reached regarding the cause of the disaster; however, it was determined that the possible causes of failure of the tank were improper design, use of steel unsuited to pressure and temperature conditions in such a tank, or a flaw in the plates or on the welding.

Procedure for Testing Self-Contained Breathing Apparatus

Development of new types of self-contained oxygen or compressed-air breathing apparatus made advisable the revision of the specifications under

19/ Berger, L. B., and McGuire, L. H., Observations on the Use of a Diesel Freight Locomotive through a Railway Tunnel: Bureau of Mines Rept. of Investigations 3887, 1946, 20 pp.

20/ Elliott, M. A., Seibel, C. W., Brown, F. W., Artz, R. T., and Berger, L. B., Report on the Investigation of the Fire at the Liquefaction, Storage, and Regasification Plant of the East Ohio Gas Co., Cleveland, Ohio, Oct. 20, 1944: Bureau of Mines Rept. of Investigations 3867, 1946, 44 pp.

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which the Bureau tests such equipment for approval for use in irrespirable and poisonous gases. Tests were made of recently developed types operating by use of oxygen-generating chemical charges, by compressed-air supplies, and by compressed oxygen. Suitable tests and standards of performance were established which all types of apparatus must meet for approval, and a revised schedule was published.21/

Single-Shot Blasting Units

The arrangements and specifications under which tests are made for Bureau approval of blasting units used in gassy mines undergo modification as new devices, and blasting practices are developed. A revised schedule for testing and approving single-shot units was published.22/

Coal-Mine Health Hazards

Laboratory and field investigations of health conditions in anthracite and bituminous-coal mines were carried out in connection with requirements of the Federal Coal Mine Inspection Act by Health Division personnel. Dust and gas samples were collected and analyzed.

Petrographic examinations and qualitative and quantitative X-ray diffraction determinations were made on gross samples of rock, coal, traction material, and rock-dusting material in bituminous coal mines. Surveys were made of unhygienic atmospheres in anthracite mines. The drilling speed and dust dissemination of electric-auger, pneumatic rotary, and pneumatic piston drills using different types of drill steel and bits were studied.

Coal mine inspectors and Safety Division personnel were trained in determination of unhygienic dust in coal mines. The course included sources, formation, properties, dissemination, and behavior of dusts; methods of sampling, determining composition and concentration of dust in air, and counting dust particles; physiological effects of exposure to dusts and permissible limits, and principles used in controlling dust.

A report was prepared23/ describing the cells or containers used to hold a known small depth of the dust-containing liquid from impinger, filter paper, and other samples collected from the air while the particles revealed by a microscope are counted to obtain an index of the concentration of dust in the sampled air. Information is given on such subjects as the kinds of cells available, kinds used, actual depths of cells, and construction of single, inexpensive cells.

21/ Procedure for Testing Self-Contained Breathing Apparatus for Permissibility: Bureau of Mines Schedule 13C, 1946; Federal Register July 17, 1946, vol. 11, No. 138.

22/ Bureau of Mines, Single-Shot Blasting Units: Schedule 12D, 1945; Federal Register, Dec. 11, 1945, vol. 10, No. 241.

23/ Brown, C. E., Beatty, R. L., and Kirby, T. B., Dust-Counting Cells: Bureau of Mines Inf. Circ. 7331, 1945, 9 pp.

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A study[24] was made to obtain information on the approximate size of the smallest dust particles revealed by or the limit of visibility of various microscopic systems. The information was desired mainly for use in interpreting and evaluating results of number-concentration and particle-size determinations on samples of unhygienic particulate matter collected from industrial atmospheres and in developing better methods for such determinations.

A series of tests[25] was made with a Traube's stalagmometer for thin liquids to see if it could be used for determining the concentration of wetting agent in solution as it came from the nozzle of the water spray used in dust-control work. This determination is important, as under similar conditions and presumably the same concentration of wetting agent in solution it was observed that the effect was not uniform - in some instances the wetting agent added seemed to have no more effect than plain water. This was probably due to improper mixing, which resulted in a concentration lacking in uniformity.

The results indicate that Traube's stalagmometer, which is commercially available, is a device that can be used for accurately determining the concentration of wetting agent in solution. Determinations can be made quickly, on the job, by a person who is not especially trained in laboratory technique. The instrument should find wide application in industry to help interpret results and control concentration wherever a wetting agent is used in a water solution.

Use of Explosives and Gaseous Explosions

Increase in Charge Limit of Permissible Explosives

At the request of the coal-mining industry seeking means of increasing coal production, the Bureau of Mines undertook an investigation to establish whether larger charges than the existing limit of 1.5 pounds of permissible explosives could be used with safety. Since then a continuing study has been conducted in the Experimental Coal Mine to determine the factors that affect the ignition of gas and dust in a coal mine using larger weights of explosives. By 1945 the results were so encouraging that tentative safe conditions were established under which permissible explosives may be used in quantities up to 3 pounds per shot. These were set forth in a new permissible schedule.[26] The tentative approval to raise the limiting charge

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24/ Brown, C. E., and Feicht, F. L., Size of Smallest Dust Particles Revealed by Various Microscopic Systems: Bureau of Mines Rept. of Investigations 3821, 1945, 11 pp.

25/ Harmon, J. P., Wetting-Agent Concentration in Water Solution Determined by the Drop-Number Method: Bureau of Mines Inf. Circ. 7351, 1946, 6 pp.

26/ Bureau of Mines, Procedure for Testing Explosives (Including Sheathed Explosives) and Blasting Devices for Permissibility and Suitability: Schedule 1F, Approved January 20 and March 14, 1945, 15 pp.; Duplicated from Federal Register, vol. 10, No. 26, Feb. 6, 1945, pp. 1476-1483; No. 60, March 24, 1945, p. 3131.

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of permissible explosive per shot hole from 1.5 to 3 pounds has been extended to December 31, 1947. Before making this approval permanent, it is desirable to acquire more knowledge of all the factors that affect the ignition of gas-air mixtures at the coal face by the flame or by the products of detonation of explosives.

To this end, 125 further tests were made in the Experimental Coal Mine with a high-rate gelatin-type permissible explosive. The explosive cartridges used were 1-1/2 inches in diameter and 8 inches long. They were loaded to the rear of boreholes of 2-1/4-inch diameter and fired by a No. 6 detonator at the rear of the charge. Under each set of test conditions, five successive tests were made to determine the probability of ignition of the gas mixture near the test face. Two principal series of tests were made. In one series, termed blow-out shots, the explosive was file-loaded; i.e., the cartridges were laid end to end in the borehole, which was not stemmed. In this case the flame and products of the detonation were ejected chiefly along the borehole. One of the objectives of these tests was to determine the effectiveness of sheathing upon the explosive cartridges. The sheath is 1/4 inch thick and covers the full length but not the ends of the cartridge; its purpose is to cool the flame and combustion products. The tests revealed that in a 36 inch-long borehole the maximum charge of unsheathed explosive for no gas ignitions in five tests was 1.4 pounds, whereas even 3 pounds of sheathed explosive (maximum weight that could be loaded to collar of borehole) gave no ignitions.

The purpose of the second series of tests was to study the danger involved in shot-firing in coal beds where the boreholes intersect fissures or cracks through which flame from the explosive might reach gas near the coal face. In this work, artificial fissures in the form of circular and rectangular holes were introduced between the borehole (which was stemmed) and a shear cut 6 inches distant from the borehole. The following factors were investigated: (1) Effect of shape and cross-sectional area of fissure; (2) effect of location of fissure in relation to explosive charge in borehole; (3) effect of weight of charge, and (4) effect of sheathing on the probability of gas ignition. It was found that with fissures of 6-inch length there is little difference in the probability of ignition in the presence of round or circular holes and rectangular holes of different shapes but of equal cross-sectional areas. This was true for file-loaded explosives and also for a loading density of one; i.e., when the cartridges were deformed so as to fill the entire cross section of the borehole. Study of the effect of increase in cross-sectional area of fissures disclosed that the probability of ignition of the gas by the explosive increases approximately linearly with increase in fissure area. To investigate the effect of location of the fissure relative to the charge in the borehole, tests were made with a round fissure (1-1/2 inch diameter) intercepting the charge at the base of the detonator, midway in the charge, and at the front of the explosive charge. The results indicate that the ignition probability increases slightly as the intercept of the fissure moves toward the front of the charge.

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To determine the effect of weight of explosive charge upon ignition tests were made with fissures of constant shapes and areas. Preliminary data indicate that with file loading and also with density-one loading, change in charge weight from 1-1/2 to 3-1/2 pounds did not increase the probability of ignition. This factor is being studied further.

To test the effectiveness of sheathing upon explosive cartridges fired in boreholes in fissured coal, tests were made with file-loaded charges of 1-1/2 and 3-1/2 pounds in the presence of rectangular fissures. The results indicate that sheathing on cartridges reduces the probability of gas ignition considerably below that of unsheathed explosives, but it does not completely eliminate the hazard. The conclusion reached from the work done thus far with shots fired by permissible explosives in stemmed boreholes in fissured coal is that the presence of cracks of fissures greatly enhances the chance of accidental ignition of gas near the working face.

Effect of Sheaths upon the Gaseous Products from Permissible Explosives

The continuing development of safer explosives for use in mining coal is of great interest to both the manufacturer and the user of permissible explosives. Tests by the Bureau of Mines and by testing stations abroad have shown that the ignition hazard of explosives fired in the presence of gassy atmospheres may be reduced by the use of a sheath. The introduction of sheathed explosives in Belgium in 1914, the adoption of sheaths by Great Britain in 1933 and by Germany in 1938, and the continuing consumption of sheathed explosives attests the importance of sheathed explosives in Europe. Although in this country sheathed explosives are not used widely, interest in them is shown by passage of State laws permitting their use on shift and by patents granted to manufacturers of explosives. The formation of poisonous gases in the detonation of explosives also may present an important hazard if they are produced in sufficient quantity. A recent study of the effects of sheaths upon the production of the poisonous gases, carbon monoxide and nitrogen oxides, and upon the total volume of gaseous products has been published.27/ Some effects of sheaths upon nitrogen and carbon recovery and upon the oxygen condition as determined in the Bichel gage and C-J apparatus are discussed here.

In the Bichel gage, the gases are produced by detonation at low loading densities without stemming. The C-J apparatus permits tamping and the use of stemming, affording a higher degree of confinement.

Nitrogen balances were calculated for the gaseous products from a large number of permissible explosives. The nitrogen recovery, which is the percentage of nitrogen in the original explosive which appears in the gaseous products of detonation, gives some indication of completeness of reaction of the nitrogen-bearing constituents, a low nitrogen recovery being indicative of incomplete reaction. Low nitrogen recovery is often, though not

27/ Fieldner, A. C., and Brewer, R. E., Annual Report of Research and Technologic Work on Coal, Fiscal Year 1945: Bureau of Mines. Inf. Circ. 7352, 1945, 103 pp.

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always, associated with high production of oxides of nitrogen. The nitrogen-recovery results (fig. 12) indicate that detonation of unsheathed explosives in the Bichel gage was essentially complete, but that the addition of a sheath often caused marked decrease in nitrogen recovery - as low as 56 percent in one case. In the C-J apparatus, nitrogen recoveries were somewhat lower for unsheathed explosives, but the addition of a sheath did not cause so marked a decrease in nitrogen recovery as occurred in the Bichel gage. In other words, explosives under confinement are not so adversely affected by sheaths as are unconfined explosives.

Carbon balances are indicative of the completeness of reaction of the carbonaceous material for unsheathed explosives. Tests in both the C-J apparatus and Bichel gage indicate essentially complete reaction for the unsheathed explosives tested (fig. 13). With sheathed explosives, carbon recovery may be more than 100 percent, because in the computation of the carbon input it was assumed that only the carbonaceous combustible material of the explosive and the sheath entered into the reaction and that none of the sodium bicarbonate of the sheath was decomposed. Some decomposition of the inorganic material did occur, as shown by the high carbon recoveries in some tests. With sheathed explosive in Bichel-gage tests, the recoveries ranged from 64.6 to 125.5 percent; in the C-J apparatus the recoveries ranged from 79.1 to 121.8 percent. These results indicate that a sheath may cause incomplete reaction of the carbonaceous material.

The oxygen-requirement ratio, which is defined as the ratio of the oxygen required to completely oxidize all the carbon and hydrogen in the gaseous products to the oxygen required to oxidize all the carbon and hydrogen in the original explosive, is also indicative of the completeness of reaction. Incompleteness of reaction or the formation of free carbon is indicated by an oxygen-requirement ratio of less than 100 percent. The oxygen-requirement ratio results are presented in figure 14. These results again indicate that the addition of a sheath may cause incomplete reaction, particularly in the Bichel gage.

Effects of Type of Ammonium Nitrate upon the Gaseous Products from Sheathed Permissible Explosives

Tests were made with two permissible explosives, sheathed and unsheathed, similar in all respects except that one sample contained spray-process ammonium nitrate and the other contained graining-kettle ammonium nitrate, to determine the effect of type of ammonium nitrate upon the production of oxides of nitrogen.

It was found that addition of a sheath caused appreciable increase of oxides of nitrogen with the graining-kettle ammonium nitrate, but no significant increase was produced for the spray-process nitrate.

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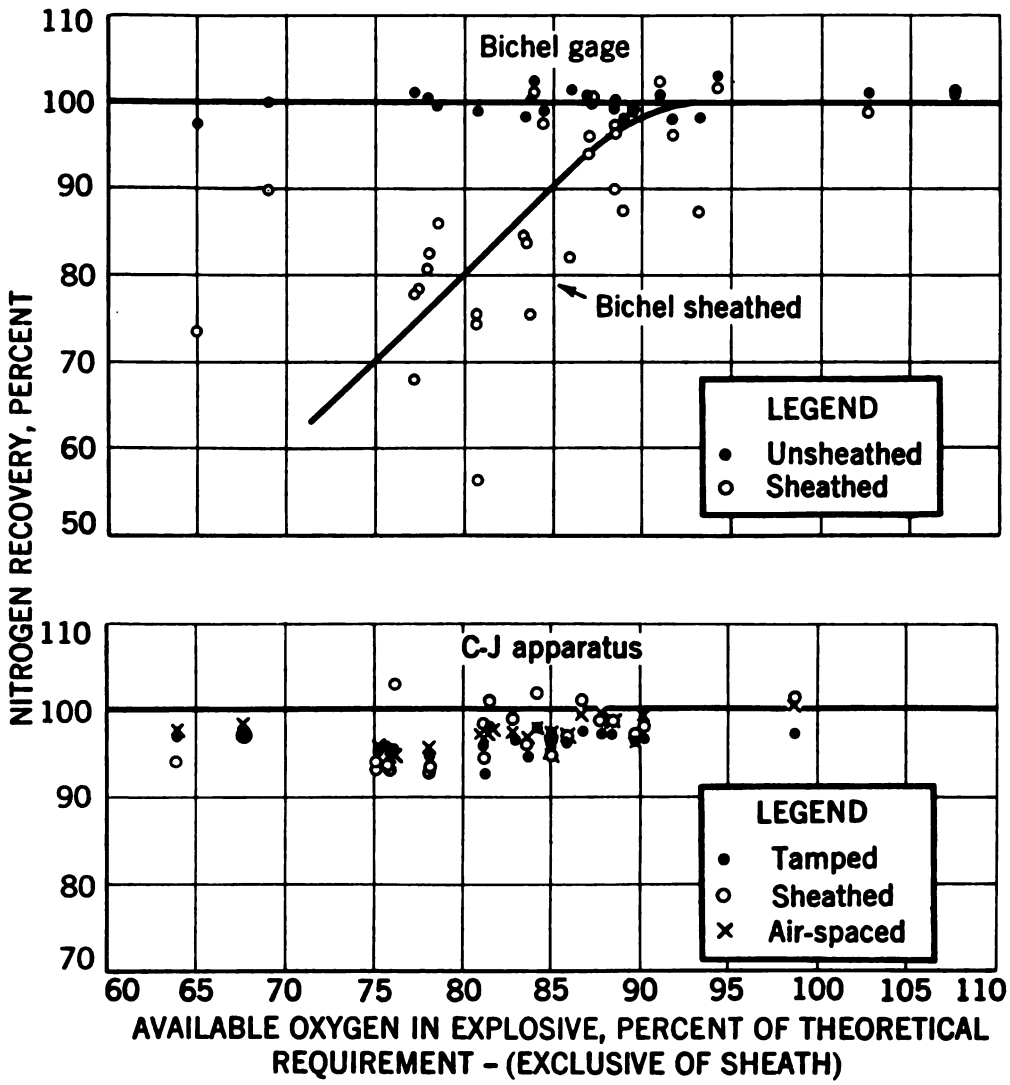

Figure 12. - Nitrogen recovery in Bichel gage and C-J apparatus.

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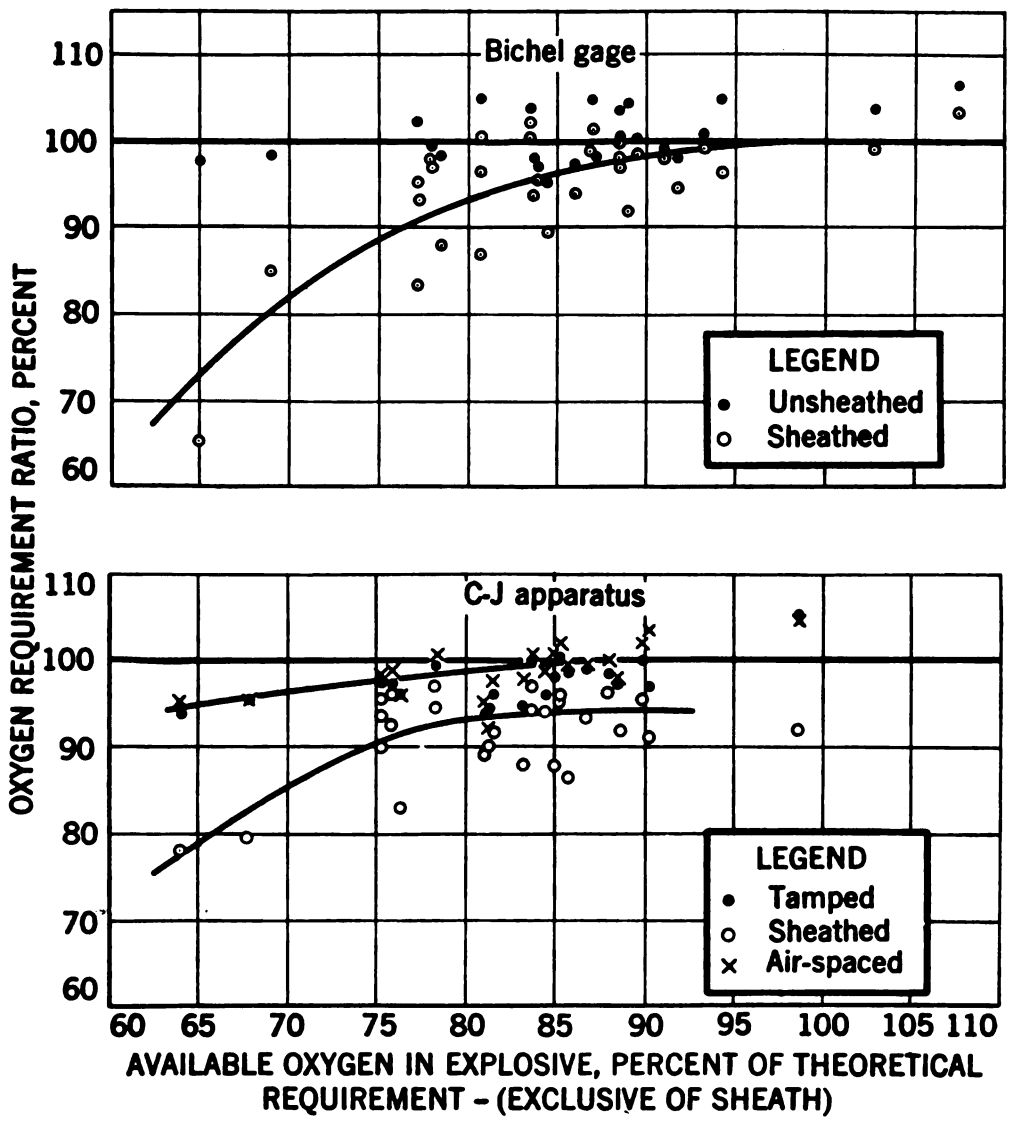

Figure 14. - Incompleteness of reaction as shown by the oxygen requirement ratio.

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Sensitivity of Explosives to Initiation by Electrostatic Discharges

It has long been recognized that there are static electrical hazards associated with the manufacture, handling, and use of explosives, particularly initiating explosives, but only within recent years has a systematic attempt been made to obtain quantitative data on such hazards. The Bureau of Mines has made and is continuing to make an extensive study of the static electrical hazards associated with the manufacture, handling, and use of explosives. Preliminary investigations on a number of explosives have been made.[28] The results indicate that a considerable number of explosives, including primer, initiator, detonator, igniter, tracer, and incendiary types may be ignited by electrostatic discharge energies less than those that may be built up on and discharged from the human body. It was established that electrostatic energies accumulated on the body may readily reach values as high as 0.015 joules, and this value was accepted as a standard in determining for what explosives extra precautions must be taken against electrostatic discharges. This standard was accepted by the U. S. Army Ordnance in drafting their safety requirements for Ordnance plants manufacturing or handling explosives.

Further work on this subject has included investigations of electrostatic sensitivity of most commercial detonators manufactured in the United States. Marked differences in sensitivity have been found, depending on composition of ignition charges and on geometry of bridge wire and case of the detonator.

List of Permissible Explosives

A permissible explosive is one that is similar chemically, physically, and in all other respects to a basic sample that has passed certain required approval tests of the Bureau of Mines. After an explosive or blasting device has passed all the required tests,[29] it may be placed on the permissible list. This list is constantly being revised owing to additions of explosives and blasting devices that have passed the tests and to transfers between the active and inactive lists. As of June 30, 1946, the list contained the names of 180 explosives and 9 blasting devices, compared with the names of 178 explosives and 9 blasting devices on the lists as of June 30, 1945.[30] Of the two new explosives added to the list, one was submitted for complete tests for permissibility and the other was an additional

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28/ Brown, F. W., Kusler, D. J., and Gibson, F. C., Sensitivity of Explosives to Initiation by Electrostatic Discharges: Bureau of Mines Rept. of Investigations 3852, 1946, 8 pp.

29/ Bureau of Mines, Procedure for Testing Explosives (Including Sheathed Explosives) and Blasting Devices for Permissibility and Suitability: Schedule 1F, approved January 20, 1945, 15 pp. (Test conditions and procedure to follow in making application for tests are given.)

30/ Tiffany, J. E., and Gaugler, Z. C., Active List of Permissible Explosives and Blasting Devices Approved Previous to December 31, 1945: Bureau of Mines Rept. of Investigations 3910, 1946, 20 pp.

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name given to an explosive already on the list. The nine models of Cardox blasting devices remained the same as last year.

Consumption of Permissible Explosives

In blasting coal, only permissible explosives should be used; otherwise disastrous explosions may occur. Permissible explosives were first introduced in 1909, and their subsequent use has increased steadily. During the fiscal year 1946, the consumption of permissible explosives in coal mines amounted to almost 91 million pounds. In addition, there were used almost 34 million pounds of black blasting powder and slightly over 97 million pounds of high explosives other than permissibles. Thus, about 2.7 times as much permissible explosives as black blasting powder was used. There has been a steadily increasing ratio since the fiscal year 1940, the first year in which the consumption of permissible explosives exceeded that of black blasting powder.

Subsequent to the introduction of permissible explosives in 1909, more than 1-1/2 billion pounds of these explosives have been used in coal mines. When used in a permissible manner, they have never been known to be the cause of a mine explosion. In the few instances of explosions in which permissible explosives were involved, the fact has been established that the explosives were used in a nonpermissible manner. Even in recent years some mine disasters have been caused by the use of black blasting powder in mines that were definitely gassy. There can be no excuse for continuing the use of black blasting powder in coal mines. The conditions of use prescribed by the Bureau of Mines form an essential part of the requirements for permissibility, and failure to observe any of these conditions constitutes a violation of the requirements and renders the use of the explosive or blasting device nonpermissible.

Field Samples of Permissible Explosives

To continue to be permissible, an explosive or blasting device must be so manufactured that it will possess all the chemical and physical characteristics of the basic sample. To check these characteristics, samples are taken in the field and retested. Tolerances that are allowed when comparison is being made with a basic sample are set forth in Schedule 1F. These tolerances define the limits beyond which field samples cannot vary and still be considered permissible for use in coal mines. Thirty samples were collected during the fiscal year. Only one failed to pass gallery test 1, and all the other samples passed both gallery test 1 and 4, although 13 of these samples did not meet the tolerances for their chemical and physical characteristics. Safeguarding the quality of permissible explosives through field sampling has been an important factor in promoting safety in the coal-mining industry in the United States.

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Miniature-Cartridge Test for Detonators

The miniature-cartridge test[31] was developed as a means of evaluating the relative initiating efficiency or strength of a detonator. This value is measured by the weight of sand crushed when the detonator under test is fired in a miniature cartridge of an insensitive mixture like TNT and iron oxide weighing 5 grams embedded in a weighed quantity of standard Ottawa sand and held in a bomb. From this weight of sand crushed is subtracted another weight of sand which is crushed when the same kind of detonator is fired embedded in a 5-gram charge of pure iron oxide. The value thereby derived represents the initiating efficiency of the detonator in comparison with values obtained from detonators with various weights of mercury fulminate used as the base charges.

Based upon the more recent tests, it is believed that the initiating efficiency of a detonator is improved (1) by substituting for a mixture of mercury fulminate-potassium chlorate (80-20) any one of the following compounds used as base charges (improvement follows in the order enumerated: (a) Tetryl, (b) PETN, (c) Cyclonite; (2) by using a reinforcing capsule for enclosing the priming charge; (3) by use of an aluminum shell instead of a gilding metal shell, and (4) by use of a small-diameter shell.

During the war, a large variety of detonators captured with enemy equipment was examined and tested. The detonators included those of German, Italian, and Japanese make. Most of the German detonators were loaded in aluminum shells charged with a base constituent of tetryl or PETN and had a priming charge of lead azide-lead styphnate enclosed in a reinforcing capsule.

Technical Assistance and Services to Others

Assistance was given to the War and Navy Departments and other Federal agencies. This included consultations on matters pertaining to demolition explosives and equipment, safety inspections of the testing area of a military reservation, surveillance tests of propellants and explosives, and testing of a sample of red phosphorus for its sensitivity to friction on the Bureau of Mines' pendulum friction device.

The Electronic Chronoscope

The Bureau of Mines is frequently consulted for recommendations on methods of improving performance and safety of explosives and for advice on explosives technology in general. This often requires short-term miscellaneous investigations. Such miscellaneous projects have included work on performance and safety of explosives using hydrogen peroxide as the oxidizing agent, liquid-oxygen explosives, and tetranitromethane-gasoline

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31/ Grant, R. L., and Tiffany, J. E., Factors Affecting Initiating Efficiency of Detonators: Ind. and Eng. Chem., vol. 37, July 1945, pp. 661-6.

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mixtures; work on methods of measuring rates of detonation and evaluation of hazards associated with a number of specific operations in industry. One such investigation led to the development of an electronic chronoscope to determine velocities of detonation of explosives that is capable of measuring time intervals as small as 1 microsecond.[32] This instrument is small, portable, and direct-reading and can be operated from stations quite remote from the explosive without transmission-line difficulties. It can be operated from the ordinary 110-volt AC circuit or can be adapted to battery operation. It can measure velocities of detonation over lengths as small as 1 centimeter of highly brisant explosives with an accuracy of approximately 5 percent. Figure 15 is a photograph of the instrument.

Ignition Temperatures of Compounds in Air and Oxygen

An apparatus consisting of a 50-pound, electrically heated, stainless-steel block, in which was enclosed a 125-cc. Pyrex Erlenmeyer flask as the ignition chamber, was used to determine the ignition temperatures of 21 different compounds that were determined in air and oxygen. This is a modification of the standard nitrate bath apparatus and served to avoid the hazards of flying glass when ignition occurred. The flask within the steel block simulates black-body conditions. The usual drop method was employed. Where the compound is a gas at ordinary temperatures, it was first cooled to the liquid state and the required amount was dropped into the apparatus. Minimum ignition temperatures determined in this modified apparatus and given in table 4 are usually only a few degrees lower than those obtained in the standard nitrate bath apparatus.

TABLE 4. - Ignition temperatures

	Formula	Minimum ignition temperatures, °C.	
		In air	In oxygen
Ethane	C_2H_6	516	506
Propane	C_3H_8	493	468
1-3-butadiene	C_4H_6	418	335
n-butane	C_4H_{10}	408	283
iso-butane	C_4H_{10}	462	319
n-pentane	C_5H_{12}	290	258
iso-pentane	C_5H_{12}	420	294
2-3-dimethylbutane	C_6H_{14}	420	298
n-heptane	C_7H_{16}	230	214
n-octane	C_8H_{18}	218	208
2-2-4 trimethyl pentane.	C_8H_{18}	434	283
Dimethyl ether	$(CH_3)_2O$	350	252
iso-phorone	$C_7H_{14}O$	462	322
n-propyl formate	$C_4H_8O_2$	455	312
iso-propyl formate	$C_4H_8O_2$	485	412
n-propyl acetate	$C_5H_{10}O_2$	457	-
iso-propyl acetate	$C_5H_{10}O_2$	476	448
1-2-dichloro-n-butane	$C_4H_8Cl_2$	276	250
Monomethylamine	CH_3NH_2	430	400
Dimethylamine	$(CH_3)_2NH$	402	346
Trimethylamine	$(CH_3)_3N$	190	175

[32] Nisewanger, C. R., and Brown, F. W., Electronic Chronoscope for Measuring Velocities of Detonation of Explosives: Bureau of Mines Rept. of Investigations 3879, 1946, 18 pp.

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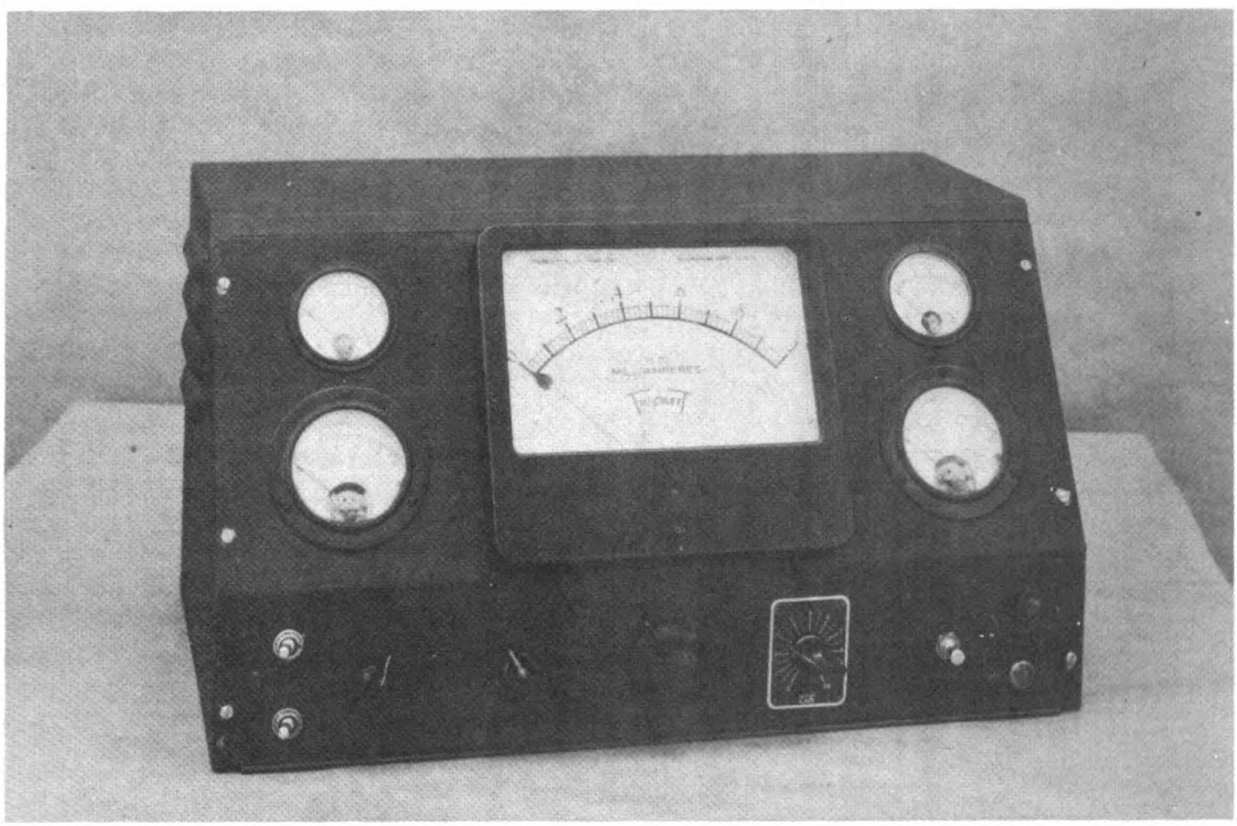

Figure 15. - Electronic chronoscope for measuring velocities of detonation.

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Limits of Inflammability of Substances in Air

Determinations of lower and upper limits of inflammability were made of a number of combustible vapors and liquids in air. Table 5 contains a summary of the values.

TABLE 5. - Limits of inflammability in air

Compound	Formula	Limits of inflammability, percent by volume	
		Lower limit	Upper limit
iso-pentane	C_5H_{12}	1.35	7.60
n-octane	C_8H_{18}	1.00	-
2-2-4 trimethyl pentane ..	C_8H_{18}	.98	5.23
Ethyl benzene	C_8H_{10}	.98	-
n-butyl formate	$C_5H_{10}O_2$	1.73	8.10
Isophorone	$C_7H_{14}O$	.84	3.80
Monomethyl amine	CH_3NH_2	4.95	20.75
Dimethylamine	$(CH_3)_2NH$	2.80	14.40
Trimethylamine	$(CH_3)_3N$	2.00	11.60

Extinction of Gasoline Flames by Inert Gases

Gasoline is one of the chief contributors to the causes of fires and explosions, which take an enormous yearly toll in property damage and cause considerable loss of life. Its widespread use makes it important to discover ways of eliminating or at least mitigating the explosion hazards of this substance. Accordingly, a study was made of the effects of various inert gases having different chemical compositions on the extinction of gasoline flames, the results of which should find considerable application.33/ The substances investigated were nitrogen, carbon dioxide, automobile exhaust gas (85 percent N_2 + 15 percent CO_2), dichlorodifluoromethane (CCl_2F_2), known as Freon-12, trichloromonofluoromethane (CCl_3F), known as Freon-11, and dichloromonofluoromethane ($CHCl_2F$), known as Freon-21.

Gasolines may have rather wide variations in chemical and physical properties. They may also contain small amounts of other substances to enhance their anti-knock properties, engine-operating characteristics, and to increase their stability during storage, transportation, and use. The results given in the report show, however, that the inflammable properties of different gasolines at normal atmospheric temperatures are influenced largely by the more volatile hydrocarbons containing 5, 6, and 7 carbon atoms in the molecule, and that the heavier constituents of higher molecular weight have a minor influence under these conditions.

33/ Jones, G. W., and Gilliland, W. R., Extinction of Gasoline Flames by Inert Gases: Bureau of Mines Rept. of Investigations 3871, 1946, 14 pp.

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Three types of gasolines were investigated having octane ratings of 73, 92, and 100 and specific gravities at 60/60°F. and vapor pressures, in mm. Hg at 25°C., of 0.7136, 0.7061, and 0.7161, and 200 mm., 182 mm., and 168 mm., respectively. The limits of inflammability of the gasolines in air, in percent by volume of gasoline vapor, were determined to be 1.50, 1.50, and 1.40 for the lower limit and 7.60, 7.60, and 7.40 for the upper limits in an open tube ignited by a flame. Slightly smaller values were found for both limits in a closed tube ignited by a spark. These results show that both limits of inflammability for the three different gasolines are almost identical, using a particular method. The average lower limit of inflammability of gasoline may be taken as 1.40 percent by volume in dry air and the average upper limit at 7.40 percent.

Results of tests with all possible mixtures of gasoline-air-nitrogen in a closed tube and with ignition by a high-voltage spark indicate that the addition of nitrogen had no appreciable effect upon the lower limit of gasoline vapor until 20 or more percent of nitrogen is added, whereas the upper limit was reduced in direct proportion to the amount of nitrogen added. At least 42.4 percent of nitrogen must be present to render all mixtures with air noninflammable.

Similar tests with gasoline-air-carbon dioxide mixtures showed generally the same results, except that the extinctive effects of carbon dioxide were found to be greater than equivalent amounts of nitrogen, as is to be anticipated qualitatively from the greater heat capacity of carbon dioxide. Only 28.9 percent of carbon dioxide is required to render all gasoline-air mixtures noninflammable.

A cheap and convenient supply of inert gas may be obtained for flame-quenching purposes by utilizing the exhaust gases from internal-combustion engines, provided the fuel-air ratio is adjusted to produce an exhaust gas containing maximum percentages of carbon dioxide and a minimum of carbon monoxide and residual oxygen. Under certain conditions, an exhaust gas containing 85 percent nitrogen and 15 percent carbon dioxide can be made, and this was used. As expected, tests with gasoline-air-exhaust gas yielded results that lie between those of nitrogen and carbon dioxide. Thirty-six percent of exhaust gas was required to be added to render all mixtures noninflammable.

Tests with the three Freons show that their flame-quenching efficiencies are much greater than other inert gases like nitrogen and carbon dioxide. Both the lower and upper inflammable limits are affected materially by additions of small percentages of these substances, causing a marked decrease in the range of inflammability. The extinction effect increases in the order $CHCl_2F$, CCl_2F_2, CCl_3F. The amounts of these substances that will render all mixtures of gasoline-air-freon noninflammable are 15.6 percent, 12.4 percent, and 11.1 percent, respectively.

It is of interest to note that all hydrocarbon-air mixtures from methane through hexane and gasoline will be free of explosion hazards at

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normal temperature and pressure if the atmosphere contains 44 percent or more of nitrogen or 31.5 percent or more of carbon dioxide.

It is sometimes more convenient to control the explosion hazards of the mixtures by means of the oxygen concentration. This is accomplished by diluting with inert gas until the oxygen concentration falls to some critical value (which varies with the inert gas added), below which the mixtures become noninflammable. These critical oxygen concentrations for the inert gases nitrogen, carbon dioxide, $CHCl_2F$, CCl_2F_2, and CCl_3F are 11.6, 14.4, 17.2, 17.6, and 17.9 percent, respectively. A comparison of the critical oxygen values for different hydrocarbons from methane to hexane and gasoline reveals that they do not differ very much for a given inert gas. Thus, when nitrogen is used, the oxygen values range from 11.0 to 12.1 percent, and if the oxygen is kept below 11.0 percent all of the indicated hydrocarbons are noninflammable in any concentration. More oxygen can be tolerated if judicious corresponding changes are made in the gasoline concentrations which the results of the investigation allow to predict.

The use of inert gases to dilute explosive atmospheres and thereby dilute the air or oxygen concentration to a point where the mixtures become noninflammable has found numerous applications in recent years. Information of the type just given has proved of great benefit to industry. By applying these data, combustible mixtures containing gasoline can be removed safely from containers and equipment, or the mixtures may be introduced into such vessels originally containing air without running the risk of explosion, provided the proper concentrations of inert gases are present.

Limits of Inflammability and Ignition Temperature of Naphthalene

Information concerning the German chemical industry and in particular the utilization of naphthalene has brought this substance to the fore as a chemical of commercial importance. Its potential fire and explosion hazards have therefore been studied.[34] The immersion method for determining the limits of inflammability of vapors was employed.[35] The limits determined are temperatures between which air saturated with naphthalene vapor is explosive when the barometric pressure is 747 mm. mercury. The lower limit was found to be 77.8°C. and the upper limit 121.8°C. Converted to concentration limits, from the vapor pressures of naphthalene at these temperatures, the lower limit becomes 0.88 percent by volume of naphthalene vapor in air and the upper limit becomes 5.9 percent of naphthalene vapor in air. The ignition temperature of naphthalene was determined by dropping a small crystal of naphthalene onto the bottom of a flask immersed in a nitrate bath. In air, the ignition temperature was found to be 587°C.; in oxygen it was 560°C.

34/ Jones, G. W., and Scott, G. S., Limits of Inflammability and Ignition Temperatures of Naphthalene: Bureau of Mines Rept. of Investigations 3881, 1946, 5 pp.

35/ Jones, G. W., Inflammation Limits and Their Practical Application in Hazardous Industrial Operations: Chem. Rev., vol. 22, No. 1, Feb. 1938, pp. 1-26.

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Effect of Pressure upon the Explosibility of Mixtures of Acetylene with Water Vapor, Air, and Hydrocarbons

Information on the explosibility and ease of ignition of acetylene is important in connection with investigations seeking to determine the cause of acetylene generator explosions. Preliminary tests made by this Bureau showed that certain sizes of carbide may, under certain conditions, attain excessively high temperatures when acetylene-water vapor mixtures are passed through carbides for extended periods.[36] Amendment 3, June 28, 1944, to Federal Specification P-C-101 was effected, which permits a maximum of 7 percent of "1/4 by 1/12" carbide by weight to pass through a 0.0661-inch sieve, and a maximum of 2 percent to pass through a 0.0555-inch sieve. Although this improvement in the size range of the carbides has aided in the prevention of explosions, explosions have occurred when conditions of the latest Federal Specification have been met. Present investigations by the Bureau of Mines indicate that one of the major causes of the explosions is the high reactivity of certain carbides with acetylene-water vapor mixtures. The results of this work will be described in a future Bureau report.

The suggestion has been made that a possible cause of explosions in medium-pressure acetylene generators is air that enters the generator during abnormal operations or remains in the carbide hopper and other parts of the generator following charging or purging. Studies of the effect of small volumes of air upon the ignition temperature of acetylene at varying operating pressures of the generators show[37] that the introduction of up to 10 percent of air into an acetylene generator does not appreciably increase the ease of ignition of the mixtures.

Further investigations have been carried out to provide data on the explosibility of acetylene containing varying percentages of water vapor, air up to 30 percent, and hydrocarbon gases at pressures from atmospheric to 15 pounds per square inch above atmospheric, which is the maximum operating pressure of medium-pressure acetylene generators.[38] The tests were carried out in a special bomb. Observations were also made on the rate of pressure rise and maximum pressure produced in an explosion. Different concentrations of water vapor were obtained by adding water and varying the temperature of the bomb. The following results were found: Acetylene becomes explosive at or above a pressure of 5.9 pounds per square inch. On adding water vapor, the pressure required to explode the mixture increases roughly proportional

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36/ Jones, G. W., Scott, G. S., Kennedy, R. E., and Huff, W. J., Explosions in Medium-Pressure Acetylene Generators: Bureau of Mines Rept. of Investigations 3755, 1944, 20 pp.

37/ Jones, G. W., and Kennedy, R. E., Effect of Pressure on Ignition Temperature of Acetylene and Acetylene-Air Mixtures: Bureau of Mines Rept. of Investigations 3809, 1945, 6 pp.

38/ Jones, G. W., Kennedy, R. E., Spolan, I., and Huff, W. J., Effect of Pressure on the Explosibility of Acetylene-Water Vapor, Acetylene-Air, and Acetylene-Hydrocarbon Mixtures: Bureau of Mines Rept. of Investigations 3826, 1945, 17 pp.

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to the volume of water vapor present. At 14.5 percent or more of water vapor, it becomes impossible to explode such mixtures below a pressure of 15 pounds gage, the maximum operating pressure of the generators. Tests made with mixtures of acetylene and air to which varying amounts of water vapor have been added show that the presence of up to 10 percent air in acetylene exerts a stabilizing effect upon the acetylene, in that they permit higher pressures to be employed (wet or dry) without danger of explosions than pure acetylene will permit. The presence of 15 or more percent of air in acetylene, however, renders the mixtures more liable to explosions than pure acetylene, the hazard increasing as the percentage of air is increased. For example, dry mixtures containing 25 to 30 percent of air can be exploded at atmospheric pressure. The effect of added water vapor is to decrease the hazard of explosion by increasing the permitted pressure.

The effect of hydrocarbons upon the stability of acetylene was investigated. The hydrocarbons selected were natural gas, propane, and butane, as they are easily procurable, cheap, and in rather wide use at present. All three had a marked stabilizing effect, better than steam and increasing in the order natural gas, propane, butane. The percentages of hydrocarbon required to render the acetylene-hydrocarbon mixture incapable of causing explosions below 15 pounds per square-inch gage are 13.8, 9.2, and 8.4 in the above order. The effectiveness thus seems to increase with molecular weight of the hydrocarbon. This information is valuable and offers a means of preventing explosions in medium-pressure acetylene generators. Propane is particularly suitable for use, as it is readily available, has a higher heating value than equal volumes of acetylene, and is marketed in convenient steel cylinders at relatively low pressures. Tests carried out at the Bureau of Mines with acetylene containing 10 percent propane in a 10-pound experimental acetylene generator, in which exceedingly high temperatures often develop in the test carbide in the hopper, have not resulted in any explosions to date. The use of hydrocarbon gases for the prevention of explosions in acetylene generators will be influenced by the difficulties encountered in introducing the required volumes of hydrocarbons into the generated acetylene and the usefulness of such mixtures in present welding and cutting equipment.

The pressures produced by the various acetylene and acetylene-air mixtures were somewhat erratic and appeared to be due to an inherent characteristic of acetylene. In general, the highest pressures resulted when the initial pressure was 12.5 pounds gage or higher. Pressures well over 200 pounds per square inch were developed, and the rate of pressure rise is extremely rapid. Water vapor reduces the maximum pressure developed. Addition of air to acetylene, other conditions being the same, does not markedly affect the maximum pressures. Up to 10 pounds gage, the addition of air appears to increase the maximum pressures, whereas at 12.5 pounds gage and above the addition of air to acetylene has only a minor effect.

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Extinction of Flames by the Addition of Helium

Tests were carried out to determine the efficiency of helium as a flame-quenching gas. The results show that this gas has about the same quenching value as nitrogen on alkyl chloride-air mixtures in the lower range of inflammability but has slightly greater quenching value than nitrogen in the upper range.

In tests with heptane-helium-air mixtures it was found that 43.5 percent of helium in heptane-air mixtures is sufficient to prevent propagation of flame under all conditions of use at normal temperatures and pressures.

PREPARATION OF COAL

Metallurgical Coal from High-Sulfur Coal

The rapid depletion of the high-quality metallurgical coal in the Pennsylvania-Pittsburgh seam has necessitated investigation into the large areas of virgin coal in the southwestern portion of the State. The coal in this area is known to be high in sulfur, although information has been lacking for completely appraising these reserves. This study was undertaken to determine the nature of the sulfur present and the possibility of reduction to a point where the clean coal would meet the limitations at present imposed on metallurgical coal by the steel industry.

It was discovered that, even within small areas, both the total sulfur and the forms of sulfur show wide variation. In the high-sulfur samples, a large proportion of the sulfur is in the organic and finely disseminated pyritic forms, which indicate that little improvement could be made by washing - at least, by high-gravity washing. Bench sampling showed that only the lower portion of the seam really responds favorably to washing.

Efficient separation at very low gravities (about 1.30 specific gravity) would produce an acceptable product after thorough blending of the washery feed. The yield, however, would be only about 50 percent, and, owing to the technological difficulties of controlling the gravity, practical operation must await the development of new and improved techniques. Fine-crushing of the coal before separation brings about no substantial reduction of sulfur unless the coal is crushed to 28-mesh top size, which would impose a serious operating problem on the cleaning plant.

Until the development of a washing process that will operate efficiently at a gravity in the 1.35 range, the only practical means of using this coal for metallurgical purposes calls for separate handling of coal from those sections that produce coal of low float-sulfur content at a commercial washing gravity. It appears that relatively large areas of this coal can be marked off and, by means of frequent sampling, a controlled raw product can be fed to the cleaning plant.39/

39/ Fraser, Thomas, and Crentz, William L., Washing Characteristics of the Pittsburgh Coal in a High-Sulfur Area in Green County, Pa.: Bureau of Mines Tech. Paper 689, 1946, 85 pp.

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Electrostatic Treatment of Fine Coal

An investigation was made into the separation of ash and fusain from the Pittsburgh-seam and the Illinois No. 6-seam coals. Samples of coal were run through the electrostatic separation of the Ritter Products Co., Rochester, N. Y. The results of this study cannot yet be considered to be indicative of the efficiency of this method of coal cleaning, but the test results of the samples are submitted.

	Ash, percent	Sulfur, percent	Fusain and inert opaque matter, percent
Pittsburgh seam			
Raw 10 x 100-mesh feed	8.57	1.73	5.0
Cleaned coal	4.22	1.43	3.2
Refuse	14.68	1.98	7.6
Illinois No. 6 seam			
Raw 10 x 100-mesh feed	26.69	3.94	5.2
Cleaned coal	22.01	3.94	1.8
Refuse	31.00	3.83	8.6

In the case of the Pittsburgh-seam coal, about 40 percent of the raw 10 x 100-mesh feed was rejected as refuse, whereas approximately 50 percent of the Illinois coal was rejected.

The results of these two tests are not encouraging as far as the reduction in ash and sulfur is concerned, but they do warrant consideration of this process as a means for the reduction of fusain and inert matter, which cannot be removed by conventional specific-gravity separation.

Preparation Characteristics of Maryland Coals

An intensive study of the preparation characteristics of the thinner coal seams of Maryland was undertaken. Although much of the preliminary work has been completed during the fiscal year, test data have not been finally analyzed and interpreted. Indications are that the seams underlying the Pittsburgh seam are all high in ash and sulfur but differ widely in their amenability to washing.

Coal-Preparation Practice in Western Europe

A survey was made of coal-preparation practices and techniques in western Europe, especially enemy countries, primarily to obtain information on any special processes that might be of value to the coal industry in America.

Performance data and operational information were obtained at plants in Germany where especially low-ash coal had been prepared during the war for the manufacture of carbon electrodes and for synthesis of liquid fuels.

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A preliminary report of these investigations was presented at the annual meeting, February 1946, of the American Institute of Mining and Metallurgical Engineers by H. F. Yancey and Thomas Fraser entitled "Coal-Preparation Practices in Germany and the Netherlands."

Washability of Coals from the Matanuska Field, Alaska

Research on coal preparation carried out during the war at the two coal mines in Alaska closest to the vital Aleutian Islands chain was published during the fiscal year.[40] The coals dealt with were from the Eska and Evan Jones mines, the two principal operations in the Matanuska field. Located about 70 miles north of Anchorage, both mines are served by a branch line of the Alaska Railroad, and both produced about 6,000 tons of washed coal per month under wartime demand. The data obtained in the washability examinations in the laboratory and by actual washing tests in the washeries at the mines were factors of vital importance to the operators at the time the studies were made and will prove useful to any other operators in this field. Moreover, because the quality of the coal renders the washing operation very difficult, owing to the presence of a high proportion of both heavy impurities and material of intermediate density, the results should prove useful to operators elsewhere who are forced by decreasing reserves to mine coal high in ash or who seek to reclaim coal from washery refuse or waste dumps.

The character of the two coals, from the standpoint of washability, is illustrated in table 6. With coals of ordinary specific-gravity composition, the correlation between washability characteristics and washery performance is sufficiently well-established to permit fairly accurate prediction of the results obtainable in washing. However, the literature contains virtually no information on the results obtainable in washing a material containing such a high proportion of impurity as that present in the Eska coal. Consequently, detailed evaluation was made of the performance of the Eska washery. This correlation between washability characteristics and washing results will be found useful by those concerned with washing unusually dirty coals mined in other localities.

Results Obtained in Washing

Three test runs were made with the jig, the only variable being the specific gravity at which the separation between coal and refuse was made, that is, the proportion of refuse removed. Each test lasted for a full shift of washery operation, and samples of the feed, washed coal, and refuse were collected at uniform intervals during the entire period.

40/ Geer, M. R., and Yancey, H. F., Washability Characteristics and Washing of Coals from the Matanuska Field of Alaska: Bureau of Mines Rept. of Investigations 3840, 1946, 17 pp.

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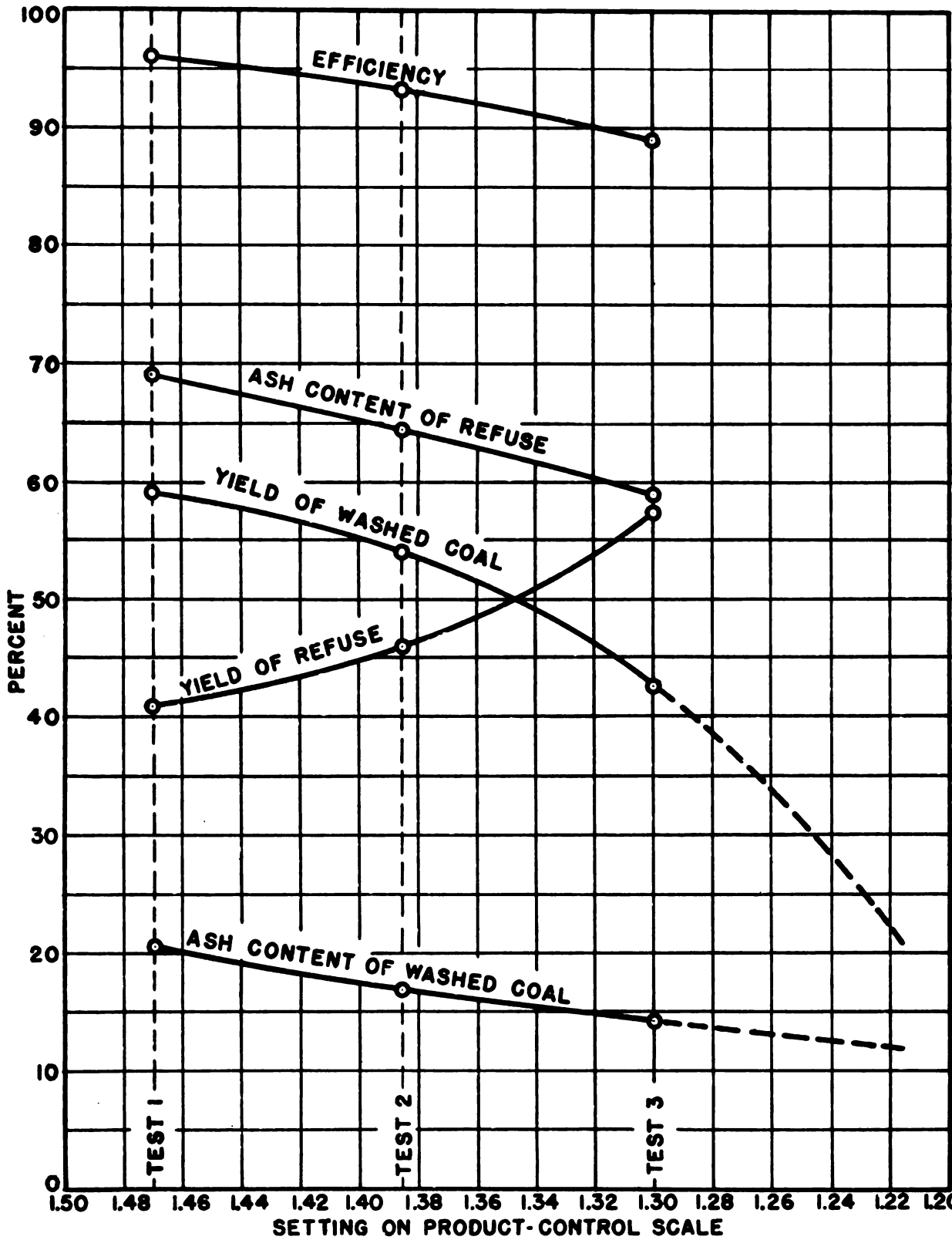

Figure 16. - Yield, ash content, and efficiency in relation to settings on product-control scale of jig.

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TABLE 6. - Specific-gravity analyses of 3-inch slack from the Eska and Evan Jones mines, Alaska

Mine	Specific gravity	Weight, percent	Ash, percent 1/	Cumulative Weight, percent	Cumulative Ash, percent 1/
Eska	Under 1.30	20.7	3.8	20.7	3.8
	1.30 to 1.40	13.0	13.1	33.7	7.4
	1.40 to 1.50	11.4	25.9	45.1	12.1
	1.50 to 1.70	14.5	39.3	59.6	18.7
	Over 1.70	40.4	72.1	100.0	40.3
Evan Jones	Under 1.35	35.5	5.6	35.5	5.6
	1.35 to 1.50	25.8	21.0	61.3	12.1
	1.50 to 1.70	17.0	38.5	78.3	17.8
	Over 1.70	21.7	62.6	100.0	27.5

1/ Moisture-free basis.

The principal results of the three washing tests are shown graphically in figure 16. The ash content of the washed coal, its yield, the ash content of the refuse, and the efficiency of the separation made by the jig between washed coal and the refuse are plotted against graduated settings on the product-control scale of the float mechanism that actuates the refuse gate of the jig.

In test 1, the ash content was reduced from 40.3 percent in the feed to 20.4 percent in the washed coal by rejecting a refuse product amounting to 40.9 percent of the feed and containing 69.0 percent ash. The efficiency of the separation was 96.1 percent; that is, the jig recovered 96.1 percent of the coal of 20.4 percent ash present in the feed. The separation was made at a point corresponding to about 1.82 specific gravity. Considerable material heavier than 1.70 specific gravity remained in the washed coal, particularly in the finer sizes. Very little low-density coal was lost in the refuse.

Tests 2 and 3 were made at successively lower specific gravities, 1.66 and 1.55, respectively, by rejecting larger percentages of refuse. The washed coals produced were substantially lower in ash content; and, of course, more coal was lost in the refuse. The efficiencies obtained in this series of tests, namely, from 96 to 89 percent, are lower than those that can be attained in washing ordinary coals.

These results show that with Matanuska coals a product of comparatively low ash content can be obtained only at a great sacrifice in yield. Thus, the value of lower-ash coal must be sufficiently great to justify the waste of natural resource involved.

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Removing of Extraneous Material from Industrial and Domestic Coals

Further progress has been made towards perfecting the new Bureau of Mines process for cleaning and dewatering the fine sizes of coal, which in the past have been mainly responsible for the slurry and sludge problems in coal-preparation plants. The cooperating company has purchased and installed two more cells, which, with the two original cells, can now be operated in series as a 4-cell machine - a combination that gives approximately three times the capacity of the original 2-cell machine.

In order to device means of improving hydraulic classification as a method of preparing the feed in the wet-table process of coal cleaning, the conditions prevailing in teeter columns of various depths have been studied. In a report on the results of this study,41/ the conditions of teeter during continuous hydraulic classification of bituminous coal, anthracite coal, and an iron-ore tailing are depicted in panorama. Data are given regarding size distribution of float-and-sink products, settling ratios, specific gravity of solids, pulp densities, and pulp consistencies of various strata within the teeter column.

The washability characteristics of Rhode Island graphitic anthracite have been studied. A chapter giving the results of this study has been written and is to be included as a part of a later Bureau of Mines report on the results of a wartime survey of the economic possibilities of Rhode Island anthracite. The washability study showed that the Rhode Island material of the type under investigation cannot be improved materially by washing unless it is first crushed to a very fine size. Liberation of ash-forming impurities is not appreciable in sizes coarser than 14 or 20 mesh.

Hand Preparation of Coal

Manual cleaning and sizing of coal at the mine have been common practice the world over, and even with the great gains that have been made in mechanical preparation practice, hand picking is still a very important operation. Yet very little has been done to systematize the application of this old-time operation or even to ascertain its effectiveness.

In the course of cooperative work with the Department of Mineral Production in Brazil to increase wartime production in that country, an exceptional opportunity was afforded to study the performance and cost of extensive hand-preparation operations.

In the State of Santa Catarina it has been the standard practice to prepare coal for railroad locomotive and industrial use by hand sorting and breaking. With the development of the national steel industry dependent upon

41/ Coe, G. Dale, Fold, I. L., Williams, M. F., Jr., and Coghill, W. H., Continuous Hydraulic Classification: Constitution of the Teeter Column Throughout its Depth: Bureau of Mines Rept. of Investigations 3851, 1946, 8 pp.

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that field for metallurgical fuel, an entirely new and different preparation problem developed, and a modern Baum jig-Rheolaveur plant has been installed to take care of it. However, the hand-picking operations have been tentatively incorporated into the general plan to remove the more obvious impurities at the mines, which are rather widely scattered and have varying rail hauls to the preparation plant.

To examine the rather clear-cut problem of economy of operation in this situation, an extensive series of tests of hand-picking performance and cost was undertaken at individual mines in the Cresciuma Urusanga area.42/

Typical data on performance and operating costs at two of the plants examined in this investigation are presented in tables 7 and 8. The picking operation is on a piecework basis at $3.50 cruzeiros per ton of picked coal in both cases (at present rate of exchange, the cruzeiro is equivalent to approximately 5 cents).

TABLE 7. - Results obtained in two typical check studies of cleaning

Product	Weight		Impurities, percent	Ash, percent
	Tons	Percent		
Bainha plant:				
R.O.M. (raw)	751	100.0	44.6	36.7
Picked coal	408	54.3	35.9	32.3
Refuse	143	19.1	80.1	54.6
Slack (raw)	200	26.6	45.0	36.5
Mina do Mato:				
R.O.M. (raw)	1,508	100.0	46.3	37.0
Picked coal	912	60.8	35.8	32.7
Refuse	326	21.6	72.3	47.8
Slack	270	17.9		43.9

TABLE 8. - Comparative operating-cost data unit costs in cruzeiros per metric ton of picked coal

Operation step	Bainha	Mato
Picking operation only	3.50	3.50
Screening		2.00
Transporting coal, slack, refuse to stock		2.00
Inspections	.19	.93
Supervision	.38	.20
Total cost	4.07	8.63
Metric tons picked coal per picker per hour	.34	.13

42/ Fraser, Thomas, Abreu, A. P., Hand Preparation of Coal in Southern Brazil: A.I.M.E. Tech. Pub. No. 1884, Feb. 1946, 12 pp.

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The operation is favored by a low labor rate and a large proportion of refuse in the raw product affording simple opportunity for effective reduction in refuse and saving in handling charges by the initial cleaning operation.

Even with these favorable conditions, preliminary hand picking does not justify itself economically, principally because of loss of coal (presumably recoverable in part if transported to washery) in the refuse.

However, as a means of preparing railroad fuel, the method appears to be particularly adapted to this field because it results in a product admirably adapted to hand firing so far as size is concerned.

Also, the burning characteristics of the fuel are substantially improved, probably more than would appear just by examination of the ash content. Although there is generally a reduction of 4 percent of ash by hand picking, this involves removal of about 10 percent of noncombustible refuse, which greatly improves the practical burning characteristics of the fuel.

STORAGE OF COAL

Information on coal storage and consulting service on coal-storage problems were given various industries and government establishments. Studies are now being made of approximately 11,000 field experiences in storing different kinds of coal.

Storage of Low-Rank Fuels

Subbituminous coal can be stored successfully in open pits if precautions are taken to minimize entrance and circulation of air. During 4 years of observations on methods for storing subbituminous coal, involving the handling of about 30,000 tons of slack coal, it was demonstrated that storage in large open pits is feasible, and the loss of heating value of the coal is as low as 1.5 percent in 1 year.43/

When coal is stored in several benches 2 to 3 feet thick in a pit made either of concrete or earth, the only entrance of air to augment heating or to support spontaneous combustion is from the top of the pile. If the coal is compacted as each bench is placed, a resistance to flow of air is set up, and thereafter the rate of air entering the coal is not rapid enough to support spontaneous combustion, and the rate of oxidation is low. As air enters the coal by diffusion, it combines with the coal to form CO_2, but some oxygen is absorbed directly by the coal without forming gaseous products of reaction, and it adds to the weight of the coal. The result is a slight increase in weight and a corresponding decrease in heating value. If the entrance of air is impeded further by a cover of fine coal or a veneer of asphalt, the coal is immersed in a water-saturated atmosphere of

43/ Goodman, John B., Parry, V. F., and Landers, W. S., Storage of Subbituminous Slack Coal in Open Pits: Bureau of Mines Rept. of Investigations 3915, 1946, 37 pp.

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CO_2 and N_2. No degradation can occur unless there is slight oxidation resulting from air entrained by changes in temperature and barometric pressure.

Practical tests of these theories were made in cooperation with the Great Western Sugar Co., which was interested in finding a safe way to store coal without immersing it in water. Each year this company stored 150,000 to 200,000 tons of coal under water. However, some of the pits have developed leaks and can no longer retain water without excessive maintenance costs. Storage trials were made over a 3-year period to determine the best procedure for placing and retaining the coal for periods of about 8 months.

In 1942, a small concrete pit was used to conduct preliminary studies on the behavior of coal in dry storage. Two hundred tons of subbituminous slack coal was placed in 3-foot benches and compacted by leveling and raking. No troubles developed, and there were no significant changes in physical or chemical properties.

During 1943, 13,000 tons of coal was placed in 4-foot benches in dry storage in a large pit at Brush, Colo., where it was observed periodically to determine behavior and condition. A few local hot spots developed as the result of errors made in placing the coal, but, again, there was no serious trouble. It was observed during this storage trial that almost all oxygen beneath the coal surface disappeared after a few weeks and the coal was enveloped in inert gases. A few hot spots developed near the surface, and these were traced to areas where the coal was segregated and not properly compacted. A few hundred pounds of coal was lost that year, and the average heat loss due to oxidation was estimated to be 1.0 to 2.0 percent.

The same pit was used the following year, but greater care was exercised in cleaning the empty pit and in compacting the coal in 3-foot benches. About 15,000 tons of coal was stored in the pit, and a bulk density of 56 pounds per cubic foot of coal was attained by careful leveling and packing of each bench. This coal remained in storage for 8 months without trouble. Periodic observations were made to determine the temperature history and the composition of gases within the coal. It was generally concluded that the procedure employed was adequate for safe storage of low-rank coal.

Several characteristics of the storage of subbituminous slack coal in open pits were observed. Self-heating is accelerated during the first 3 weeks of storage, and the temperature rises at a rate of 2° to 3° F. per day until a temperature of 100° to 130° F. is reached. Thereafter, the temperature tends to level off and to decrease until the coal is removed. It is indicated that during the first 3 weeks of storage the rate of oxidation is relatively rapid; however, the reaction slows as the concentration of oxygen diminishes. The temperature in the coal is highest near the surface, where air enters by diffusion, and decreases with the depth of the coal. Products of oxidation, consisting of approximately 12 percent carbon dioxide, 87 percent nitrogen, traces of oxygen, and other gases, fill the void spaces of the pit after a few weeks in storage, and thereafter losses by oxidation are minimized. About 45 percent of the oxygen passing into

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the coal mass is absorbed by the coal, and the net decrease in heating value is 1.0 to 2.0 percent over a period of a year.

In storing subbituminous coal in open, dry pits, the following general points should be considered:

1. The walls of the storage pit should be tight, preferably made of concrete or excavated in dense earth or clay.

2. It is important to avoid segregation of sizes in the coal as it is being unloaded or placed in the pit. The accumulation of zones of fine and coarse coal provides air entry courses, which will supply oxygen to the interior of the stored coal. The entire mass of stored coal should be as homogeneous as possible.

3. In placing the coal in the pit, care should be taken to store it in shallow benches 2 to 3 feet in depth. Observations have shown the latter to be both practical and economical. Each bench should be leveled and compacted to as high a packing density as is practical. A bulk density of 56 pounds per cubic foot can be attained readily with slack coal.

4. It has been demonstrated that uniform compaction of the pit should be practiced, taking care of the marginal edges as well as the inner areas. Remnants of old coal remaining from the previous storage should be spread out in thin, uniform layers over the bottom of the pit before benches of new coal are placed in storage. Observations have revealed that snow, weeds, and other debris that have accumulated on the bottom of the pit or on benches of coal should be removed before any new coal is placed, as they preclude dense packing and eventually become loosened void spaces, which enable air to circulate.

5. The finished stored coal should be level in appearance and, if possible, flush with the top edge of the pit walls to avoid entrainment of wind currents. Valleys and peaks appearing on the surface of the storage pile should be leveled, because irregularities will catch wind currents and will induce air circulation to cause self-heating.

6. In a pit with tight side walls, air can enter only through the top surface. To impede movement of air into the coal mass, the surface should be compacted by rolling or scraping to break down the top few inches of slacked coal into fines.

UTILIZATION OF COAL

Combustion

Fuel-Engineering Service

Fuel engineering service to government establishments in the inspection, selection, and use of fuels and fuel-burning equipment was continued. At

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the request of the Veterans Administration, total operating cost comparisons were made when using oil, gas, and coal, and the type of fuel-burning equipment was recommended for 38 new projects.

At the request of the War Department, a fuel engineer was sent to the Ninth Service Command, West Coast Area, to study the operating conditions of the heating plants of that area. Vancouver Barracks, Barnes Hospital, Fort Lawton, Port of Embarkation, Vancouver General Hospital, Baxter General Hospital, and Fort Lewis, all in the State of Washington; Camp Adair, Oregon; Fort Douglas, Utah; Chico Army Air Field, Camp Beale, and installations in San Francisco and Sacramento, California, were visited. At many plants he found that the boilers were running at the extremely low ratings of 25 to 30 percent. The ratings were increased with resulting improvement of efficiency of about 10 percent. In one instance, an extreme case at Baxter General Hospital, coal consumption was reduced 50 percent, saving 65 tons per month. Boiler-room instruments were corrected and put in operation, keeping of daily records was begun, stokers were adjusted, and operators were trained.

At the request of Howard University, the possibility of their purchasing part of their electric current was studied; consideration was also given to the possibility of enlarging the present heating plant to provide the necessary heat for a number of new buildings. Consulting and inspection service was given regarding the new heating plant of the Bureau of Mines at Bruceton, Pa., as to installation of boilers, brickwork and fuel-burning equipment, and type of boiler-water treatment. Consulting service as to the purchase of fuel appropriate for the equipment was given the Justice Department and Office of Indian Affairs for all of their plants and also for a number of plants of the Federal Public Housing Authority, the Veterans Administration, the War Department, and the Navy Department. At the request of the Procurement Division, recommendations were made as to specifications for Federal purchases of charcoal and coke. Consulting service on various special problems was given some 30 different government agencies.

Work was continued on the research at the power plant of the Washington Navy Yard, where unusual accumulations of deposits and corrosion on boiler tubes were occurring. As these deposits consisted largely of various types of sulfate compounds, an investigation was made as to the amount of sulfur trioxide in the products of combustion under various operating conditions. In the attempt to use the code method of the American Society of Mechanical Engineers for making this determination, it was found that satisfactory results could not be obtained. The inhibitor used in the test to prevent oxidation of the sulfur dioxide to sulfur trioxide would not maintain its effectiveness. After extended studies, important modifications of the test code method were developed that, as far as studied, permit satisfactory and reproducible results. A series of tests was then made to determine the sulfur trioxide in the products of combustion from two different types of stoker-fired boilers operating at different ratings. Calculations of these data have not yet been completed. It is planned to

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vary certain operating features of the stokers to reveal the exact operating conditions under which the deposits are at a minimum. The studies to date have made possible much better operating conditions from the standpoint of boiler outage and corrosion.

Cooperating with the Commonwealth of Pennsylvania, test equipment and procedures at the Philadelphia State Hospital, Philadelphia, Pa., were studied to determine the comparative economy of burning low-ash and high-ash anthracite barly on McClave stokers under water-tube boilers.

The cooperative work with the Air-preheater Corporation, New York City, on the study of deposits and corrosion in air preheaters was continued. Studies were made of the accumulation and type of deposits and resulting corrosion on air-preheater plates from installations in New York and New Jersey. In all cases, the deposits were acid and belonged to a family of sulfate compounds. A number of experimental plates of many different types of material were installed at a plant in Indianapolis, Ind., for the purpose of making comparative studies. Special studies are also being made at a plant in New Jersey where the air preheater is subjected to unusually high temperatures. Problems of continuous cleaning of the air preheater plates are being studied, using air and steam saturated and also superheated. Correlation of the deposits and corrosion difficulties is being made with different types of fuel and fuel-burning equipment.

National Fuel Efficiency Program

Owing to the withdrawal of funds for all war projects, work on the National Fuel Efficiency Program ceased early in the fiscal year. The work of the same general nature as described in the annual report for the fiscal year 1945 was continued for about 2 months. Although officially closed in the central office at Washington, volunteer activity in the field was continued even as late as March 1946 by individuals, on their own initiative, who considered that work on fuel conservation should be carried on in their particular areas. Considerable literature was prepared, some thirty articles appearing in the press, of which representative articles are listed.44/

44/ National Fuel Conservation and Smoke Abatement. Address by R. R. Sayers, Director, Bureau of Mines, read at the convention of the Smoke Prevention Association of America, Inc., at Columbus, Ohio, October 17, 1945, and published in Proceedings of the Smoke Prevention Association of America, pp. 15-19, incl; Solid Fuel Engineer, vol. 5, No. 5, pp. 6, 7, and 8, Feb. 1946; and Mechanization, vol. IX, No. 12, pp. 133 and 135, Dec. 1945.

Saving Fuel is Good Business. Article on the National Fuel Efficiency Program published in Heating, Piping and Air Conditioning, vol. 17, No. 11, p. 623, Dec., 1945; vol. 18, No. 1, Jan., 1946.

Barkley, J. F., Points to Remember in Operation of Boiler Plants: Solid Fuel Engineer, Oct. 1945, p. 5.

Barkley, J. F., Some 1945 Ideas About the Use of Coal for Locomotives. Proceedings, Railway Fuel and Traveling Engineers Association, 1945, pp. 134-152.

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Good cooperation was had with industrial and publishing concerns on publicity matters. Ten publishers of magazines requested complete files of the quiz sheets and expressed their intention of publishing many of them. Material was prepared for inclusion in an Illinois Central Railroad movie film on fuel conservation. A National Fuel Efficiency Program "slug" was completed for inclusion in the general advertisements of industrial concerns. A number of concerns agreed to carry the small "slug" in some appropriate corner of their advertisement.

Thousands of fuel-consuming plants were visited by volunteers throughout the United States, 15,858 signed pledges of cooperation being returned to Washington. The plants and buildings represented in this group consumed approximately 100 million tons of coal equivalent. A great number of instances of individual savings were reported to Washington, ranging from a few percent of the fuel up to some 40 percent. Based upon such reports as received, it is apparent that about 5 million tons of coal equivalent was being saved per annum during the last 12 months of the program's life. Had the program been continued under the impetus built up, it is believed that a much higher per annum saving would have accrued in the next 12 months.

Recognition of the fine work done by thousands of volunteers was given to the extent possible. Commendatory certificates were given to the members of the advisory National Fuel Efficiency Council and to scores of Coordinators heading up individual areas in the United States. A somewhat similar commendatory card was given several thousand volunteer regional engineers. A great number of individuals and organizations expressed their satisfaction in having been associated with this work, and many expressed their opinion that a national fuel-efficiency program should be a peacetime effort of the Federal Government.

Boiler Feedwater Conditioning

Analyses and resulting recommendations were made on 13,404 samples of boiler water during the fiscal year, as follows: 11,800 from the War Department; 707 from the Veterans Administration; 266 from the Office of Indian Affairs; 265 from District plants; 178 from the Department of Justice; 54 from the Public Health Service; 43 from the National Housing Agency; 43 from the Post Office Department; 19 from the Navy Department; 15 from the Department of Agriculture; 8 from the Commerce Department; 4 from the Federal Works Agency; and 2 from the National Advisory Committee for Aeronautics. Special analyses with recommendations were made on 12 samples of various types of water for the War Department, Veterans Administration, and Public Health Service. Reports and recommendations covering 15 analyses of various scales, sludges, and deposits and 7 covering analyses of boiler compounds were made for government agencies. Two hundred and thirty five special Bureau of Mines field water test kits, 12,321 bottles of chemical reagents, and 14,448 test-kit replacement items were distributed to various government activities. At the request of the District Government, an acceptance test was made on a Zeolite softener installed at Gallinger Hospital. At the request of the Post Office Department, cooperative arrangements were completed to clean condensate lines at the new city post office,

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Washington, D. C.; some of these lines are in concrete columns and are nearly filled with rustlike deposits. Consulting service was given the District Government on corrosion control in oil-fired boilers used in a number of high schools, the Public Health Service on corrosion from distilled water, and the Washington National Airport on corrosion control in high-pressure drips. At the request of the Transportation Corps Board of the Army, a study was made of a type of organic compound being used to treat locomotive-boiler water at all posts; a report was submitted to the Army explaining the action of this compound in boiler water and why it could not be so used universally. Instructions on boiler-water treatment and testing were given operating engineers at numerous government heating plants. Studies were made of the effect of boiler-water sludge on determining the values of total dissolved solids by hydrometer; on changes of the chemical composition of boiler-water samples between the time of sampling and analysis; on methods for eliminating the interference of tannin in colorimetric tests for phosphate; on the stability of stannous chloride reagents; on the determination of total dissolved solids in boiler waters by electrical conductivity; and on the determination of chloride in boiler waters containing tannin.

Boiler Water Research

Field studies were made with special corrosion-test apparatus in condensate lines, which gave further information on the corrosiveness of condensate-containing carbonic acid; it was shown that corrosive rates are satisfactorily reduced when the acidity is neutralized with an amine having proper volatility and alkalinity. The knowledge gained during the study of the physical and chemical behavior of one amine, cyclohexylamine, in the steam system has permitted material economies in using the amine treatment and has extended its application to systems for which it had previously been regarded as being too expensive. New applications will be made at many of the veterans hospitals, where the increased costs of boiler-water treatment can readily be justified in preventing failures and unscheduled outages, which cannot be tolerated in hospitals. The field studies also reveal that the fundamental relationship between corrosion rates, dissolved gases, flow rates, and temperatures are not known and that many of the prevailing concepts are not consistent with the available data. Common errors made in the evaluation of such data have been shown to result from unwarranted extrapolation and improper averaging.[45/] As a better understanding of the corrosion process and the factors involved would be expected to result in improved methods of prevention and control, laboratory investigation of the influences of several of these factors has been started.

Laboratory testing with the embrittlement detector showed that potassium hydroxide, like sodium hydroxide, could concentrate in the seams and tube ends of boilers to cause intercrystalline corrosion and cracking of

45/ Berk, A. A., Discussion of "The Evaluation of Data": Proc., Sixth Annual Water Conference, Engineers' Soc. Western Pennsylvania, 1945.

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the boiler metal.[46] This conclusion was corroborated by tension tests in which stressed test specimens were exposed to the attack of hot concentrated potassium hydroxide solutions. Therefore, the treatment of boiler waters with potassium instead of sodium chemicals is not a method of preventing boiler-metal embrittlement. However, the practical methods of preventing embrittlement trouble in waters containing alkaline sodium compounds are also effective when potassium treatment is used. The data obtained from these researches do not support a recently announced theory for the mechanism of the intercrystalline cracking of mild steel.[47] Six cases of stationary boiler cracking were reported for analysis and correlation during the fiscal year. The operating pressures ranged from 100 to 465 psi. at steaming rates up to 100,000 pounds per hour. Failures ranged in severity from a single tube end to widespread seam cracking that necessitated the replacement of the entire boiler. Embrittlement detector tests had been run in units attached to two of the boilers, and the results had shown the boiler water to be dangerous before the cracks were discovered. The accumulated results of embrittlement detector tests in almost 1,000 stationary plants have confirmed the effectiveness of nitrate treatment to prevent cracking. Treatment with quebracho has also been satisfactory in many instances, and no test specimens have failed when all the causticity in the water was replaced with the hydrolytic alkalinity of trisodium or tripotassium phosphate. A most satisfactory decrease in the number of cracks found in the boilers of Chesapeake & Ohio Railway locomotives also has resulted from the application of nitrate treatment.

Representatives of the Bureau of Mines have actively participated on A.S.T.M. committee work related to boiler-water research. A standard specification for the embrittlement detector test[48] has been prepared. Under the chairmanship of a representative of the Bureau of Mines, a subcommittee has prepared three new tentative specifications for methods of analysis, which have been adopted[49] by the A.S.T.M.

External Corrosion of Furnace-Wall Tubes

The investigation of external corrosion of furnace-wall tubes in large slag-tap furnaces, conducted in cooperation with the Combustion Engineering Co. since June 1942, has continued. The papers presented before

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46/ Berk, A. A., and Rogers, N. E., Embrittlement Cracking in Waters Containing Potassium Salts: Trans., Am. Soc. Mech. Eng., vol. 67, July 1945, pp. 329-334.

47/ Berk, A. A., Discussion of "Theory of Stress Corrosion of Mild Steel": Trans., Electrochem. Soc., vol. 87, 1945, p. 233.

48/ A.S.T.M. Designation D807-46T.

49/ Tentative Method of Test for Total Aluminum and Aluminum Ion in Industrial Waters: A.S.T.M. Designation D857-46T.
Tentative Method of Test for Manganese in Industrual Waters: A.S.T.M. Designation D858-45T.
Tentative Methods of Test for Silica in Industrial Waters: A.S.T.M. Designation D859-45T.

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the American Society of Mechanical Engineers in December 1944,[50], [51] based upon experiments at an arbitrary temperature of 1,000°F., afforded an explanation of the mechanism of attack where characteristic deposits found at the site of corrosion were the water-soluble, sulfate type, but it was fully recognized then that quantitative data were needed at lower temperatures, approaching the nominal metal temperature of the furnace tubes in high-pressure units. Accordingly, quantitative experiments have been made to determine the effect of concentrations of SO_3 from 0.10 percent to 1.0 percent at temperatures of 700° to 1,000°F. on (a) various alkali-metal sulfates, (b) iron oxides, (c) synthetic "enamel" deposits, (d) actual "enamel" deposits, (e) metallic iron, (f) various metals suitable for application to furnace tubes as protective coatings, and (g) these same metals in contact with both SO_3 and "enamels." The last item essentially was a series of corrosion tests in which the rate of attack of the respective metals was determined under conditions believed to simulate those favoring the attack on furnace tubes.

Furnace-tube corrosion associated with an external scale rich in iron sulfide and magnetite, referred to as the "sulfide" type of attack to distinguish it from the "enamel" type, does not occur as frequently as the latter, but wherever it has been found the degree of attack usually has been quite severe. Preliminary studies have been made, based upon the supposition that CO in furnace gases reduces alkali-metal sulfates to sulfides, which then react with the tube metal to form iron sulfide. Pure CO was passed over K_2SO_4, Na_2SO_4, glaserite, and $K_3Fe(SO_4)_3$ at 1,000° F., but the results to date cast doubt upon the validity of the assumed mechanism. Another approach has been to determine the effect of FeS, in an atmosphere of up to 5 percent oxygen and at various temperatures, upon the rate of corrosion of iron. It is believed that the data from these studies will provide a more rational explanation for the sulfide type of attack.

To account for local concentrations of SO_3 at the surface of furnace-wall tubes in slag-tap units that are higher than the over-all concentration of SO_3 in furnace gases, it has been assumed that when the coal ash adhering to the tubes begins to slag, sulfur gases are slowly evolved. Experiments are in progress to determine the composition and the rate of evolution of gases from several synthetic and actual coal ashes at various temperatures.

Experiments have been conducted to determine the manner in which alkali-metal sulfates are formed on furnace-wall tubes. The results to date demonstrate that alkali metal vapors in an atmosphere containing small concentrations of SO_3 will condense on relatively cool surfaces as simple or complex sulfates, depending on the source of the alkali-metal vapor.

50/ Reid, W. T., Corey, R. C., and Cross, B. J., External Corrosion of Furnace-Wall Tubes. I. History and Occurrence: Trans., Am. Soc. Mech. Eng., vol. 67, 1945, pp. 279-288.

51/ Corey, R. C., Cross, B. J., and Reid, W. T., External Corrosion of Furnace-Wall Tubes. II. Significance of Sulfate Deposits and Sulfur Trioxide in Corrosion Mechanism: Trans., Am. Soc. Mech. Eng., vol. 67, 1945, pp. 289-302.

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Laboratory and field experience with various metals applied to steel as protective coatings has produced promising results, and it is hoped to have enough confirmatory data for publication in the near future. Engineering changes in the burner arrangement of several furnaces to provide better distribution of coal and air have been responsible for the prevention of further attack and notable reduction of attack in many cases.

Smoke Abatement

At the annual meeting of the American Society of Mechanical Engineers at New York City in November, a report was presented, "Example Sections for a Smoke-Regulation Ordinance," prepared as a summary of the work for the past 5 years of the A.S.M.E. Model Smoke Law Committee, of which a Bureau of Mines representative is chairman. Nineteen written discussions were received. A special meeting of the Committee was held at Pittsburgh, Pa., in May. The 19 discussions were studied, and certain changes were made in the report. It is estimated that the report will reach its final form in the near future. At the request of the A.S.M.E., a representative of the Bureau of Mines served as chairman and prepared questions for a panel discussion of smoke abatement at the semiannual meeting at Detroit, Mich., in June. At the request of the City of Los Angeles, Calif., a meeting to discuss the abatement of smoke and noxious gases was called in Washington and was attended by representatives of the National Research Council, the Public Health Service, the National Academy of Science, the Bureau of Mines, and the mayor of Los Angeles; as a result of this conference the mayor decided upon certain administrative moves in connection with the problem. Consulting service was later given the Los Angeles area on carrying out this work. Conferences with the editorial staff of the Chicago Sun and representatives of the Bureau of Mines were held regarding a smoke-abatement campaign in Chicago. At the request of the District of Columbia government, a representative of the Bureau of Mines served as a member of a committee of three to hold hearings on the practicability of changing certain smoke-abatement regulations of the District of Columbia. Consulting service was given the cities of Wilmington, Del., and Niagara Falls, N. Y., on the establishment of smoke-abatement ordinances. Since the cessation of the war, requests for Bureau of Mines publications on smoke abatement have increased substantially.

Packaged Fuel Containing Anthracite Fines

Packaged fuels made from mixtures of anthracite fines and bituminous coals with an asphaltic binder have been investigated as a means of utilizing the smaller sizes of anthracite and of supplementing the supplies of domestic fuel.[52] In cooperation with a large industrial-equipment manufacturer, 29 experimental lots of widely varying composition, including several consisting only of anthracite and binder, were prepared on commercial coal-packaging equipment. Physical tests made to simulate the effects

52/ Schmidt, L. D., Reid, W. T., Seymour, William, and Myers, J. W., Physical and Combustion Characteristics of Packaged Fuel Containing Anthracite Fines: Bureau of Mines Rept. of Investigations 3882, 1946, 35 pp.

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of handling and storage indicated that adequate cold strength was obtained in packaged fuel made entirely from anthracite fines with moderate quantities of asphaltic binder, as shown in figure 17. Other lots made from a wide variety of blends of anthracite fines and high- or low-volatile bituminous coal with the binder also possessed adequate cold strength. The compressive strength was shown to increase as the percentage of binder increased and also as the absolute size of the coal increased. The physical stability or resistance to abrasion and spalling was found to increase with increasing binder content within the limits studied. The compressive strength decreased, however, if coal of a relatively uniform size was used, as this increased the percentage of voids in the blocks.

The burning tests were conducted with equipment and firing techniques similar to those used for household heating. Packaged fuel made entirely from anthracite with reasonable quantities of asphalt binder crumbled excessively while burning, with the result that the grates became clogged, and adequate burning rates could not be maintained with the natural draft normally available from domestic chimneys. This crumbling, resulting from the lack of adequate caking properties of the fuel mixture, also caused excessive loss of unburned combustible matter in the ashpit. The addition of sufficient bituminous coal to the mixtures resulted in the manufacture of packaged fuels with satisfactory combustion characteristics. It was found that the production of smoke from these lots of packaged fuel tested depended primarily upon the quantity of binder used and only secondarily upon the volatile content and quantity of the bituminous coal used in the mixture. The effect of increased binder content upon the quantity of smoke produced is shown in figure 18. The asphaltic binder was found to produce about eight times as much smoke as the same quantity of high-volatile bituminous coal. Packaged fuel containing anthracite fines has been produced commercially by the company that cooperated in these tests.

Utilization of Mixtures of Bituminous Coal and Anthracite

Calculations and summaries were completed of many tests at a number of plants on various types of industrial stokers of mixtures of slack bituminous and fine anthracite.[53/] These field tests show that the use of such mixtures is very practicable and that it is possible to change and usually to adjust to an improved condition the characteristics of a fuel bed of eastern bituminous slack coal by the addition of proper amounts of anthracite. The improved uniformity of the fuel bed of single-retort stokers makes possible less manual attention to the bed. Air flow through the fuel bed is affected. Smoke is decreased. In general, the fly ash as well as the unburned combustible in the fly ash and refuse are increased. Clinkering in the fuel bed is generally lessened. The peak-load carrying capacity of the stoker is definitely affected, usually adversely for the higher percentages of anthracite. In general, the efficiencies obtained on single-retorn stokers with the lower percentages of anthracite in the mixtures are

53/ Barkley, J. F., Burdick, L. R., and Wiggers, R., Tests of Bituminous-Anthracite Mixtures on Industrial Stokers: Bureau of Mines Rept. of Investigations 3916, 1946, 16 pp.

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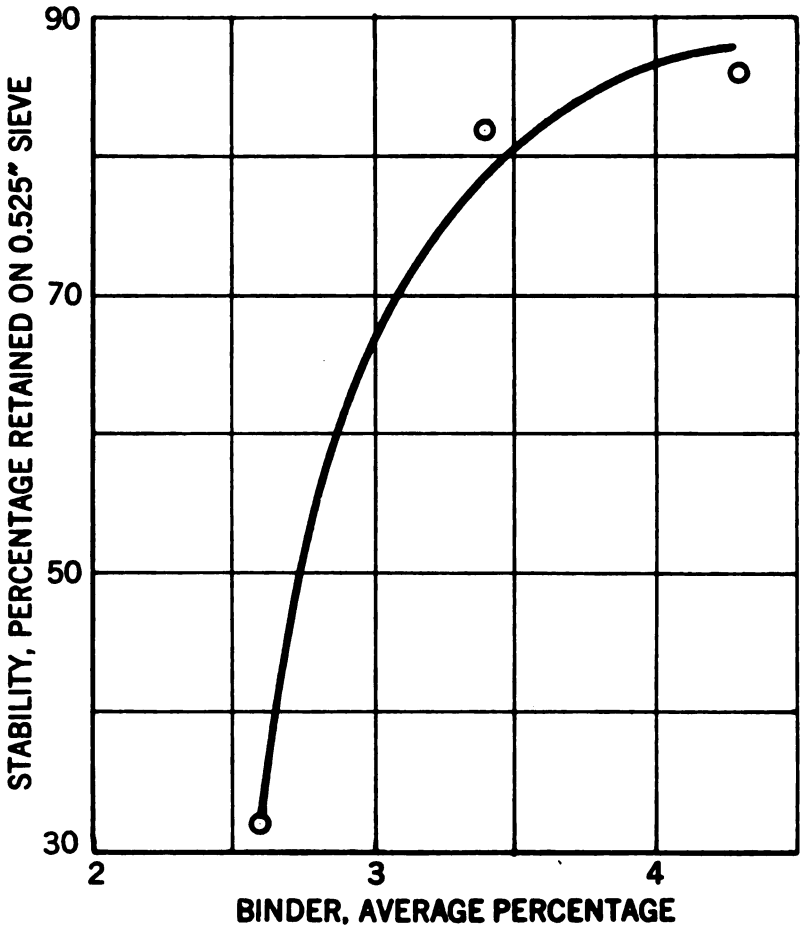

Figure 17. - Effect of binder content upon stability for packaged fuel containing only anthracite.

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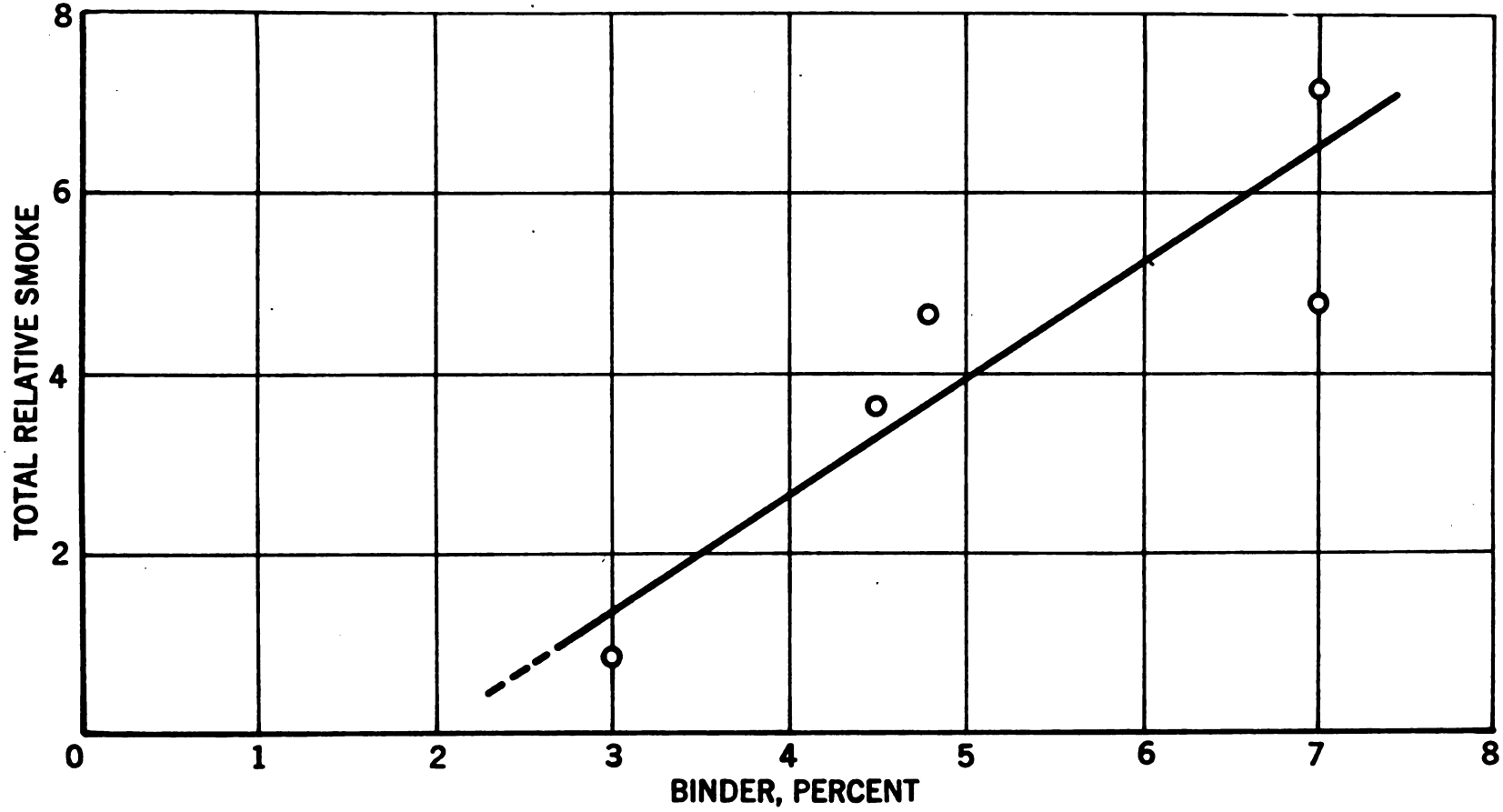

Figure 18. - Effect of binder content upon total smoke produced for packaged fuel containing only anthracite.

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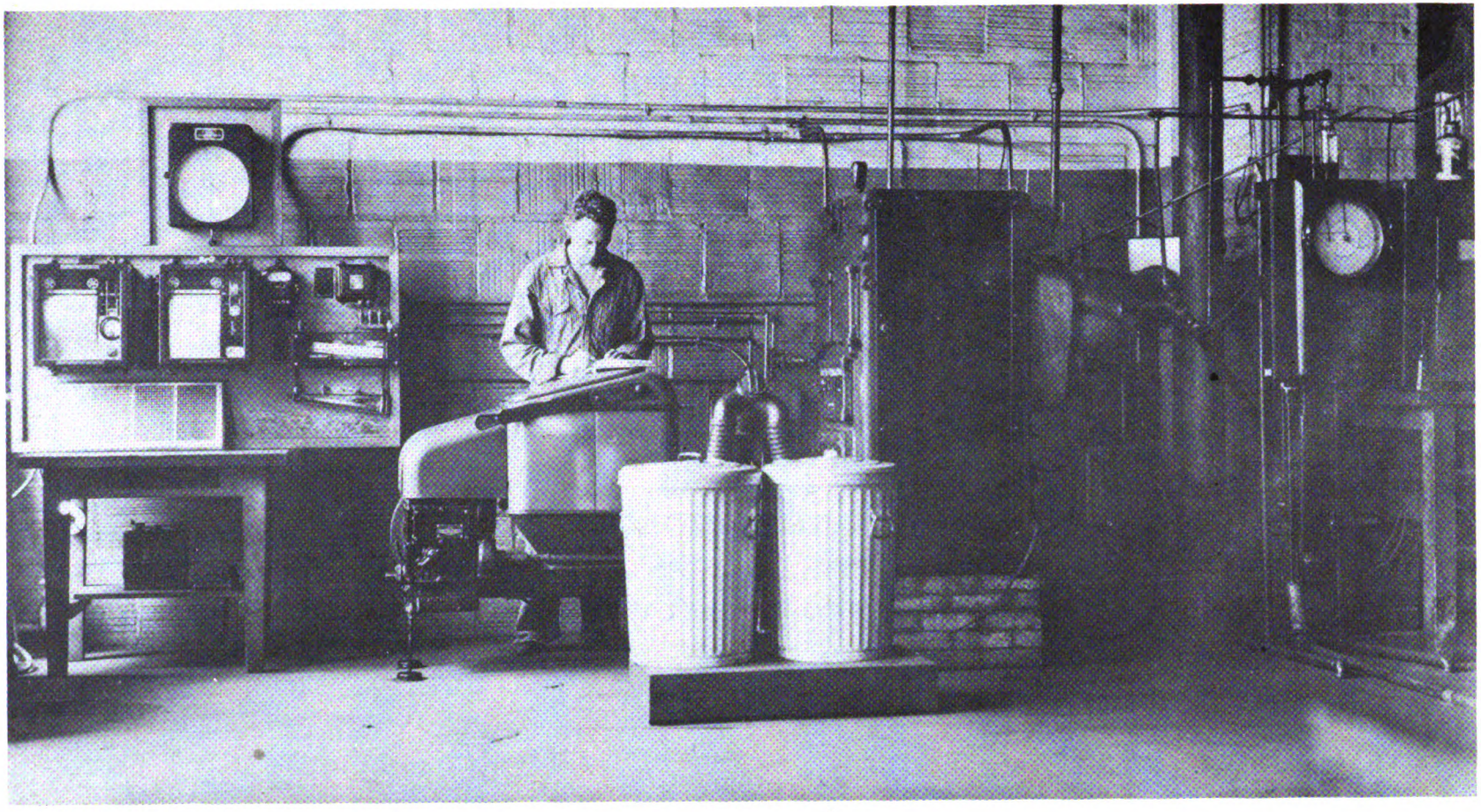

Figure 19. - Anthracite ash-removal-type stoker, hot-water boiler, and equipment used for burning trials.

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about the same as with bituminous coal only. With the higher percentages of anthracite, the over-all efficiencies were found to be somewhat lower. The best percentage of anthracite to use at a given installation depends on many factors, such as the size consist, the volatile and ash content and ash fusion of the fuels to be used, the load conditions, the smoke production, and the type of equipment. It must be determined by trial for each installation. Usually, relatively simple mixing schemes can be devised at each plant to obtain a satisfactory fuel mixture for stoker use.

Ash-Removal, Domestic-Stoker Tests

Coals vary so widely in their burning characteristics that all coals prepared for domestic stoker use cannot be burned efficiently on every type of stoker. The overfeed stoker operates satisfactorily when burning low- to high-ash, noncaking, bituminous or subbituminous coals; whereas, on the clinkering-type, underfeed, bituminous stoker, both caking and noncaking bituminous and subbituminous coals are burned. Generally, these coals have ash contents below 12 percent and ash-softening temperatures between 1,800° and about 2,600° F.

Bituminous and subbituminous coals with high ash-softening temperatures and high-ash contents have not been able to enter the domestic stoker-coal market in the Pacific Northwest, because clinkers are not formed readily on clinker-type underfeed stokers, especially in mild weather. The labor required and attendant inconvenience entailed in handling the large volume of ash are particularly undesirable. Accordingly, an investigation was undertaken to enable mines producing coals of the kind described to enter the stoker-coal market. An anthracite stoker that conveys the refuse from the ash pit of the furnace and deposits it in dust-tight disposal cans was selected for the burning trials.54/

It is thought that home owners in the East who already have such stokers will wish to learn how successfully bituminous caking coals can be burned, in case of an anthracite shortage.

Eight representative Washington coals were selected with respect to their caking properties and ash-softening temperatures, and eighteen heat-balance trials on these coals were made to determine their relative suitability for use on this type of stoker. All the coals were burned at low and medium feed rates, and, in addition, two of the coals were burned at a high feed rate.

Figure 19 shows the stoker, the hot-water boiler, and the accessory equipment, and table 9 shows the principal results of the burning trials.

54/ Yancey, H. F., and Johnson, K. A., Burning Bituminous and Subbituminous Coals on an Anthracite, Ash-Removal-Type, Domestic Stoker: Bureau of Mines Rept. of Investigations 3849, 1945, 14 pp.

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TABLE 9. - Principal results of stoker-burning trials on coals selected to show effect of caking properties and ash-softening temperatures

	Feed rate per hour, pounds	Heat balance, percent		
		Efficiency, heat to water	Ashes	Dry flue gases
Coal and caking properties:				
Roslyn-Cascade 1 and 4, fair caking	10.0	66.0	11.3	10.4
	17.7	63.0	17.8	9.2
Roslyn 5, fair caking	9.5	64.5	13.9	13.3
	15.3	64.6	11.5	14.1
Elk, poor caking	11.4	72.8	6.7	14.0
	17.2	65.0	12.0	14.1
Bellingham, noncoherent	10.1	79.4	.8	10.4
	16.1	75.1	.7	15.3
	22.1	73.2	1.1	15.6
Coal Creek, noncoherent	9.8	75.9	.9	11.5
	16.1	72.8	.6	16.0
	21.5	71.5	.7	17.9
Coal and ash-softening temperatures:				
Harris, 2,910+° F.	10.2	74.5	1.6	11.5
	16.4	73.7	1.4	13.6
Coal Creek, 2,780° F.	9.8	75.9	.9	11.5
	16.1	72.8	.6	16.0
	21.5	71.5	.7	17.9
Bellingham, 2,620° F.	10.1	79.4	.8	10.4
	16.1	75.1	.7	15.3
	22.1	73.2	1.1	15.6
Tono, 2,380° F.	10.0	77.8	1.2	8.5
	16.4	76.5	1.2	10.8
McKay, 2,280° F.	9.6	67.9	1.0	20.4
	14.9	67.7	1.1	21.8

It will be observed that caking coals are generally unsuitable for this type of stoker, but those caking only slightly may be used at some sacrifice in efficiency occasioned by the loss of unburned coke in the refuse. Thus, when emergency substitution of coal becomes necessary, weakly caking coals could be burned in anthracite ash-removal-type stokers with a reduction in efficiency of only 10 to 15 percent.

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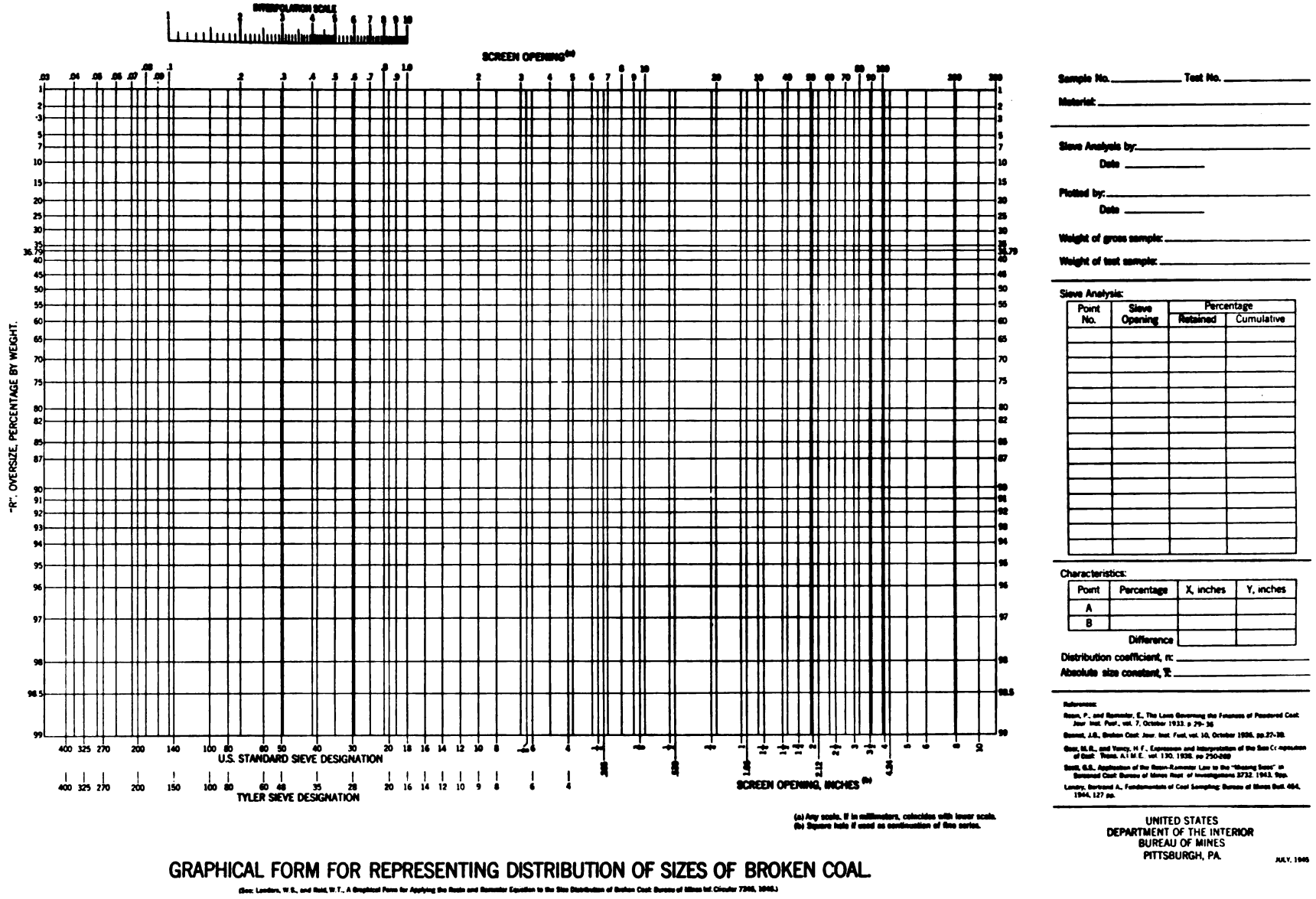

GRAPHICAL FORM FOR REPRESENTING DISTRIBUTION OF SIZES OF BROKEN COAL.

(See: Landers, W. S., and Reid, W. T., A Graphical Form for Applying the Rosin and Rammler Equation to the Size Distribution of Broken Coal. Bureau of Mines Inf. Circular 7245, 1945.)

Figure 20. - Form for applying Rosin and Rammler relationship to size consist.

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Noncaking bituminous and subbituminous coals whose ash-softening temperatures are above 2,600° F. were burned with high efficiency, showing the practicability of using anthracite stokers to burn this type of coal for house heating. Coals with ash-softening temperatures as low as 2,380° F. also were burned with high efficiency, but the noise made by the ash-removal mechanism in crushing clinkers might be undesirable in a home. Stokers of this general type are especially desirable for burning high-ash coals, because the labor and inconvenience of handling the ashes are minimized.

Analysis of Size Distribution in Broken Coal

The Rosin and Rammler equation for expressing the distribution of sizes in crushed material is finding wide application in research and industrial problems relating to the utilization of coal and coke. It can be used to estimate the quantities of material that have been removed by screening from run of mine, crushed, or slack coal; in comparing different lots of coal, coke, or other materials; and in evaluating certain physical properties of coal. As the mathematical application of this relation to size consist data is cumbersome, a convenient and accurate graphical method has been in use for this purpose. The ruling and preparation of the special form required is tedious, and a printed form has been made available in order to extend the use of this valuable means of expressing size consist, particularly in industrial control.[55/] Figure 20 shows the form together with provisions for recording the tabular data and computations required for the application of the Rosin and Rammler relationship. In analyzing size consist data by this method, cumulative percentages retained on each sieve used in the sieve analysis are plotted against the corresponding sieve size or opening. For coal, coke, or other materials that have been shown to follow the Rosin and Rammler relationship, these points will lie on a straight line if the size consist resulting from mining or crushing has not been altered by the removal or loss of any of the material. If these points lie on a curve, it is possible to calculate the quantities of material that have been removed. When these points lie upon a straight line, the location and slope of this line provide an easy means of evaluating and describing the size distribution of the sample.

The Measurement of Heat Absorption in the Primary Furnace of a Central Station Boiler

In order to obtain the operating and design data require for the most efficient utilization of fuel in central station power plants fired with pulverized coal, the Bureau of Mines is cooperating with the American Society of Mechanical Engineers in a study of the efficiency of heat absorption in the primary furnace of a large central-station boiler. The tests are being conducted on a dry-bottom, tangentially fired, pulverized-fuel unit with a rated load of 475,000 pounds of steam per hour at 1,525 pounds per square inch gage pressure. This unit, operated by a large distributor

55/ Landers, W. S., and Reid, W. T., A Graphical Form for Applying the Rosin and Rammler Equation to the Size Distribution of Broken Coal: Bureau of Mines Inf. Circ. 7346, 1946, 5 pp.

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of electrical energy, was put on the line in September 1945 and has undergone three series of tests since that time. Heat absorption in the primary furnace of this unit is being studied with respect to the following variables: (1) Age of the unit; (2) cleanliness of the walls; (3) load; (4) excess air, and (5) inclination and arrangement of the adjustable burners. The Bureau is determining the heat content of the gases leaving the furnace from measurements on the temperature, composition, and velocity of the gases at the furnace outlet. Results of these tests are then correlated with those obtained by other members of the cooperating group on the furnace-heat absorption calculated from the density of the steam-water mixture along a selected wall tube, and from the thermal gradient through the fire side of the furnace tubes, as determined by a large number of thermocouples around the furnace.

The techniques of test and the methods of calculation developed for these tests have been shown to be accurate and will be used in similar tests on other types of boilers after completion of the present series.

Automatic Water Heater

An automatic stoker-fired heating system using low-rank fuels was developed and used successfully by the Bureau of Mines.[56]

The report presents practical performance data on the service water-heating system utilizing subbituminous coal and describes the development of an improved double-tank system for heating water for domestic purposes. Over a period of about 2-1/2 years, the two systems were used to supply hot water for a small laboratory using about 50 gallons a day, and observations were made periodically to find ways to heat water automatically at maximum efficiency and at the lowest cost.

A small commercial stoker originally designed for heating service water with anthracite was improved, with slight modifications to adapt it for burning subbituminous coal. The system employed a single storage tank and a combustion dome to extract heat from the coal-burning stoker. It performed satisfactorily on subbituminous coal, but the electric-power consumption was disproportionately high, because the stoker and blower unit operated continuously.

A double-tank heating system was designed to provide additional hot-water storage capacity and to increase the available heating surface, which would extract more of the heat from products of combustion discarded in the single-tank system. The stoker-operating mechanism was changed to allow it to operate intermittently and thus take advantage of the reactivity of subbituminous coal. This improved system was operated for about 18 months and was tested periodically to determine its performance under actual conditions developed during normal use.

56/ Parry, V. F., Landers, W. S., and Goodman, J. B., Automatic Water Heating Utilizing Subbituminous Coal: Bureau of Mines Rept. of Investigations 3890, 1946, 11 pp.

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The following table compares the performance of the single- and double-tank heating systems when operating under practical conditions with subbituminous coal having a heating value of about 9,500 B.t.u. per pound:

	Single-tank system	Double-tank system
Coal burned per day ..... pounds	13.5	8.1
Service water used per day ..... gallons	47.3	37.0
Coal used per gallon of water ..... pound	.28	.22
Refuse ..... percent of coal burned	6.1	4.6
Ash in refuse ..... percent	69.0	81.5
Electric power consumed ..... kw.-hr. per day	2.0	.21
Temperature of service water ..... °F	150	165
Operating costs per month ..... dollars	3.62	.98
Coal at $6.00 per ton	1.22	.73
Power at 4 cents per kw.-hr.	2.40	.25
Cost per 100 gallons of service water heated ..... dollars	.26	.08

For several months the improved system furnished hot water at a rate of about 50 gallons a day for a total operating cost of about $1 per month, including the cost of coal and electric power. The cost of water from the single-tank system was about $3 per month owing to the disproportionately higher cost of electric power.

The improved system as operated used about 100,000 B.t.u. per day and operated at an efficiency of 71 to 72 percent. The electric power required was cut from 2.0 to 0.21 kilowatt per day by making the stoker operate intermittently. The final design of the improved system provided for a coal hopper containing 8.0 cubic feet, which was enough fuel for 1 month's operation without recharging. The ash receiver was 0.2 cubic-foot capacity and retained the ash from 300 pounds of coal.

Performance tests on the improved system revealed that water could be heated from 40° F. to 130° F. at a rate of 16 gallons an hour. Various grades of subbituminous slack coal were used in the stoker, but it was observed that the unit performed best when coal 1/16 by 1/2 inch in size was used. When using 1/2- by 0-inch slack coal, some troubles were experienced with fly ash. The stoker and heating system were designed for subbituminous coals, and the particular system employed is not suitable for higher-rank fuels because they are not reactive enough to maintain a live fuel bed unless provisions are made for frequent blowing of the bed. Subbituminous coal and lignite will remain kindled in the fuel bed for 200 minutes.

Mineral Wool from Rhode Island Anthracite

Studies are being conducted in cooperation with Rhode Island State College to determine if a slag suitable for the manufacture of mineral wool can be produced from Rhode Island anthracite. The fuel, containing more than 30 percent of high-silica ash, is mixed with local limestone and gasified

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in the slagging-type gas producer shown in figure 21. Preliminary tests have indicated that a satisfactory slag can be produced from these materials if a high enough rate of gasification can be maintained in the equipment. Because of the low reactivity of the anthracite, high temperatures must be maintained in the fuel bed, and it may be necessary to water-cool the reaction zone of the producer.

Wartime Utilization of Coal in Germany

Early in 1945, a Solid Fuels Mission was organized under the auspices of the Technical Industrial Intelligence Committee to study wartime developments in Germany coal technology. Some of these developments in the preparation and utilization of solid fuels in Germany during World War II have been summarized in published reports.57/

The mission gave particular attention to the chief coal-mining districts of Germany - the Ruhr and the Saar. As the Ruhr coal basin was the arsenal of German heavy industrial and war-making economy, more attention was given to it. The Bergbauverein, with headquarters at Essen-Heisigen, carried out the principal research on all phases of coal technology from mining to utilization. Of particular interest in the field of preparation were plants at Essen and Aachen for preparing a coal containing only 0.5 to 0.7 percent ash used in manufacturing electrodes. The plant at Essen, located at the Queen Elizabeth mine, accomplished this by a two-stage cleaning process consisting of a heavy-medium separation in a suspension of magnetite followed by pulverization of the clean product and its recleaning by froth flotation. At Aachen the extremely low-ash coal for electrode manufacture was produced by a combination of froth flotation and digestion of the recleaned coal in a hot, dilute mixture of hydrochloric and hydrofluoric acids. Another process used in the Ruhr to give virtually ashless coal involved solution of the coal in organic solvents produced by hydrogenation of coal in another plant and the separation of the undissolved material by filtration.

In the Saar, satisfactory blast-furnace coke was made from high-oxygen coking coals by substituting low-temperature coke made by the Krupp-Lurgi process for low-volatile coal previously shipped to the Saar from the Ruhr.

In both the Saar and the Ruhr, two significant features of coal preparation differed from common practice in the United States. The first was the wide use of piston jigs for treating closely sized coal, even down to one-half inch, as distinguished from the wide range of sizes treated in the Baum-type jig in the United States; the other was the almost universal practice at coking-coal mines of cleaning coal finer than about 1 mm. by froth flotation.

57/ Yancey, H. F., Mining in Europe; Wartime Coal Production and Utilization: Coal Age, 1945, vol. 50, pp. 115-118.

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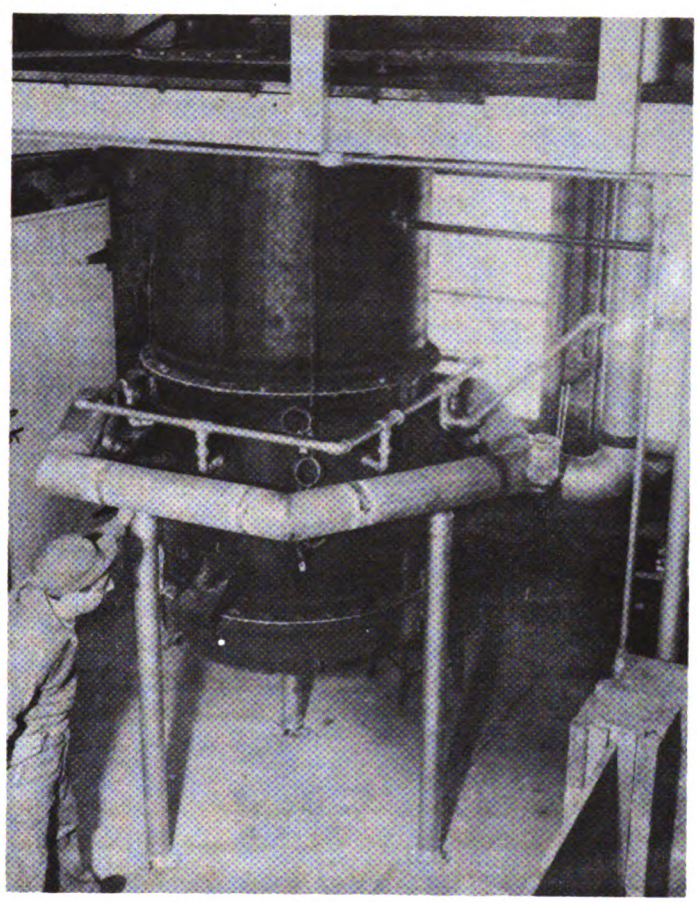

Figure 21. - Slagging-type gas producer for use on Rhode Island anthracite.

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CARBONIZATION AND GASIFICATION

Laboratory Tests of Coking Coals

In the survey of gas- and coke-making properties of American coals, small-scale laboratory carbonization tests are made of coals from various sources to determine their suitability for the production of metallurgical coke. The tests include the Fischer low-temperature carbonization assay, the United States Steel Corporation high-temperature distillation test, and the Bureau of Mines agglutinating-value test. In a study of the carbonization properties of western coals, agglutinating-value tests of 88 coals and small-scale carbonization assays of 29 coals were reported. The agglutinating value results showed that certain areas in Colorado, New Mexico, Utah, Washington, and Wyoming contain coals that have coking properties sufficiently high for the production of metallurgical coke. Low-temperature tar yields by the Fischer assay ranged from 11.5 gallons per ton of coal for a Washington lignite to 59.0 gallons per ton for a cannel coal from Utah. No trend in yields could be noted for coals from the various States because of the wide variation in rank of the coals. Cannel coal from a bench in the King-Cannel mine, Kane County, Utah, yielded 59.0 gallons of tar and 3.82 gallons of light oil per ton of coal, compared to 26.4 gallons of tar and 2.02 gallons of light oil from the bench of common banded (bright) coal in the same mine. Spores that are commonly abundant in cannel coals are prominent contributors to tar and light-oil yields.

Tar and light-oil yields of a sample of lignitic coal from Ouachita County, Ark., were determined by the Fischer low-temperature carbonization assay. On an as-received coal basis, the tar and light oil yields were 25.4 and 1.83 gallons per ton, respectively. These yields are considerably higher than those usually obtained from coals of this rank. The high tar yield of a similar coal from the same district tested several years ago was accounted for by the large amount of yellow waxy material it contained; it is probable that this also accounts for the unusually high tar yield of the more recent sample of coal.

During the year, agglutinating-value tests were made of more than 150 diamond drill-core and mine samples of coal submitted in connection with exploration projects for new sources of coking coals. The coals were from the following areas: Coosa field in St. Clair County, Ala., Coal Creek area in Paonia district, Gunnison County, Colo., Lookout Mountain field in Dade County, Ga., Georges Creek field in Allegany and Garrett Counties, Md., and Deep River field in Chatham County, N. C. The agglutinating values of the Coosa-field coals in Alabama ranged from 4.6 to 9.9 kilograms. These values, obtained at a 15:1 ratio of silicon carbide to coal, indicate that many of the coals are strongly coking. The Coal Creek coals from Colorado had agglutinating values ranging from 3.4 to 5.9 kilograms. These values are higher than those determined for Minnesota Creek coals from the same district previously examined. Agglutinating values and BM-AGA carbonization-test results showed that some of the Coal Creek coals have coking properties superior to those of the lower Sunnyside coals of Utah, a standard western coal for making metallurgical coke. The agglutinating values of the Georgia

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and North Carolina coals and most of the Maryland coals would classify them as strongly coking.

Agglutinating values that have proved to be useful as an approximate measure of coal oxidation are used to determine the changes that take place in the coking quality of coals during storage. In connection with a survey of storing properties of coals, agglutinating values of 48 samples were determined during the year. The coals examined were from the following sources: Black Creek and Hill beds, Alabama; Elkhorn No. 3 bed, Kentucky; Raton bed, New Mexico; Pittsburgh No. 8 bed, Ohio; McAlester bed, Oklahoma; Pittsburgh bed, Pennsylvania; McKay and Roslyn No. 5 beds, Washington; and Beckley, Eagle, Pocahontas No. 3, Pocahontas No. 4, and Powellton beds, West Virginia.

In connection with a study of complete gasification of Alabama coals, agglutinating values of 11 samples of coal were determined by the Bureau of Mines method. The values ranged from 3.2 to 8.3 kilograms, which indicates that the samples ranged from fair- to good-coking.

Alaskan Coals

Fischer low-temperature carbonization assays were made of five samples of Upper Cretaceous coals from northern Alaska to determine the value of these coals as a potential source of liquid fuel. The samples were collected in the summer of 1945 by U. S. Geological Survey parties working in the Chandler and Anaktuvuk Rivers area as a part of the Navy Department's petroleum investigations in Naval Petroleum Reserve No. 4. Rank classification of the coals could not be made because of loss of bed moisture during the period between collection and analysis of the samples; however, four of the coals were probably no higher than subbituminous in rank, as indicated by their high oxygen content on a dry, ash-free coal basis. The range of yields of carbonization products was as follows: Char, 66 to 79 percent; tar, 8.7 to 21.5 gallons per ton; light oil, 0.95 to 1.74 gallons per ton, and gas, 1,890 to 2,950 cubic feet per ton. The coals were noncoking in the Fischer assay; the carbonized residues were loose powders.

Chilean Coals

In connection with a survey of carbonizing properties of coals of Chile, made at the request of the Chilean Government, Fischer low-temperature carbonization assays were made of a sample of attrital coal from the Elena mine, Magallanes Province, and of samples of attrital coal and anthraxylon from the Volcano mine, Magallanes Province. These samples were separated from selected samples of lump coal to determine the effect of types of coal upon the yields of carbonization products. The low-temperature carbonization yields on a dry, mineral-matter-free basis are as follows:

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	Elena mine,	Volcano mine	
	attrital coal	Attrital coal	Anthraxylon
Coke, percent	63.1	63.8	71.3
Tar, percent	14.3	11.7	2.4
Light oil, percent	0.91	0.98	0.24
Gas, cubic feet per ton coal .	3,610	4,090	3,680
Gas, B.t.u. per pound coal ...	620	1,010	730

The attrital coals are shown to yield much more tar and light oil and less coke than the anthraxylon. These differences in tar and light-oil yields may be due to variations in the concentration of waxy material in the two types of coal. The tars from the attrital coals contained a large amount of pale-yellow waxy material similar in appearance to that from certain resinous or waxy western coals. The anthraxylon yielded virtually none of this material.

Chinese Coals

At the request of the Foreign Economic Administration in assisting the Chinese Government in the development of coal resources of China, small-scale laboratory coking tests were made of two samples of washed coal from the Nantung mine, Tolientzu bed, Szechwan Province, and three samples of coal from the Tung Ling and Wei Yuan mines, Szechwan Province.

According to their fixed-carbon content, the samples from the Nantung mine are classified as near the border line between low-volatile and medium-volatile bituminous coals. Their agglutinating values at the 15:1 ratio were 7.9 and 8.5 kilograms, respectively. These high values, together with visual examination of the carbonized residues from the laboratory carbonization assay, showed that the coals were strongly coking. Yields of carbonization products by the United States Steel Corp. high-temperature carbonization assay were as follows:

	Dry-coal basis	
	Sample 1	Sample 2
Coke, percent	84.3	82.4
Tar, gallons per ton coal	4.2	5.1
Light oil, gallons per ton coal	1.88	2.37
Ammonium sulfate, pounds per ton coal	19.4	18.4
Gas, cubic feet per ton coal	9,960	9,980
Gas, B.t.u. per pound coal	2,580	2,660

The agglutinating values of the coals from the Tung Ling and Wei Yuan mines ranged from 3.5 to 7.0 kilograms, which shows that these coals are medium to strongly coking.

Sardinian Coals

As a part of an investigation of a commercial low-temperature carbonization plant at San Antioco, Sardinia, Fischer assays were made of samples

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of lump coal from three mines in Sardinia to determine the yields of low-temperature carbonization products of these coals. The coals are high-volatile and contain 8.4 to 9.2 percent sulfur on an as-received coal basis. The following summary shows the yields of carbonization products obtained:

	Tanas mine	Serbariu mine	Bacu Abis mine
Carbonized residue, percent	68.2	67.2	66.7
Tar, gallons per ton coal	42.9	32.1	33.6
Light oil, gallons per ton coal	3.82	3.41	3.98
Liquor, percent	4.8	9.2	9.1
Gas, cubic feet per ton coal	1,980	2,100	2,030
Gas, B.t.u. per pound coal	960	820	770
H_2S in gas, percent by volume	25.6	23.1	26.3

Because of their high content of sulfur, these coals yield a large amount of hydrogen sulfide on carbonization; the percent by volume of hydrogen sulfide in the gas ranged from 23.1 to 26.3 percent. The carbonized residue from the Tanas mine sample showed that this coal is quite strongly coking. The other two coals were noncoking, or nearly so.

The Free-Swelling Index of Coal

A report[58] was prepared in response to numerous requests for information concerning the A.S.T.M. Tentative Method for Free-Swelling Index of Coal, particularly as to interpretation of results and operating techniques. It includes a discussion of some of the results that have been obtained by the Bureau of Mines, following installation of the test in 1938 and procedures used to overcome difficulties with coals giving carbonized buttons whose shapes do not correspond with the standard profiles as given in the published method.

Briefly, this swelling test consists of heating one gram of pulverized coal in a silica crucible over a gas flame under prescribed conditions to form a coke button, which is compared in size and shape with a series of standard profiles numbered 1 to 9. These profiles are intended to cover the size and shape of buttons obtained from coals covering a wide range in plastic properties. The number of the standard profile with which the maximum cross-sectional area of the button most nearly corresponds is recorded as the swelling index.

The free-swelling test has proved to be useful for obtaining information regarding the coking properties of coal on fuel beds. Coals that swell excessively may cause trouble by clogging the grates and requiring a high forces draft to obtain the proper rate of combustion. The free-swelling index test gives better differentiation between coals in measuring this characteristic than can be obtained by examination of the coke buttons from the standard volatile-matter test or from agglutinating-value results.

58/ Selvig, W. A., and Ode, W. H., An Investigation of a Laboratory Test for Determination of the Free-Swelling Index of Coal: Bureau of Mines Rept. of Investigations 3989, 1946, 8 pp.

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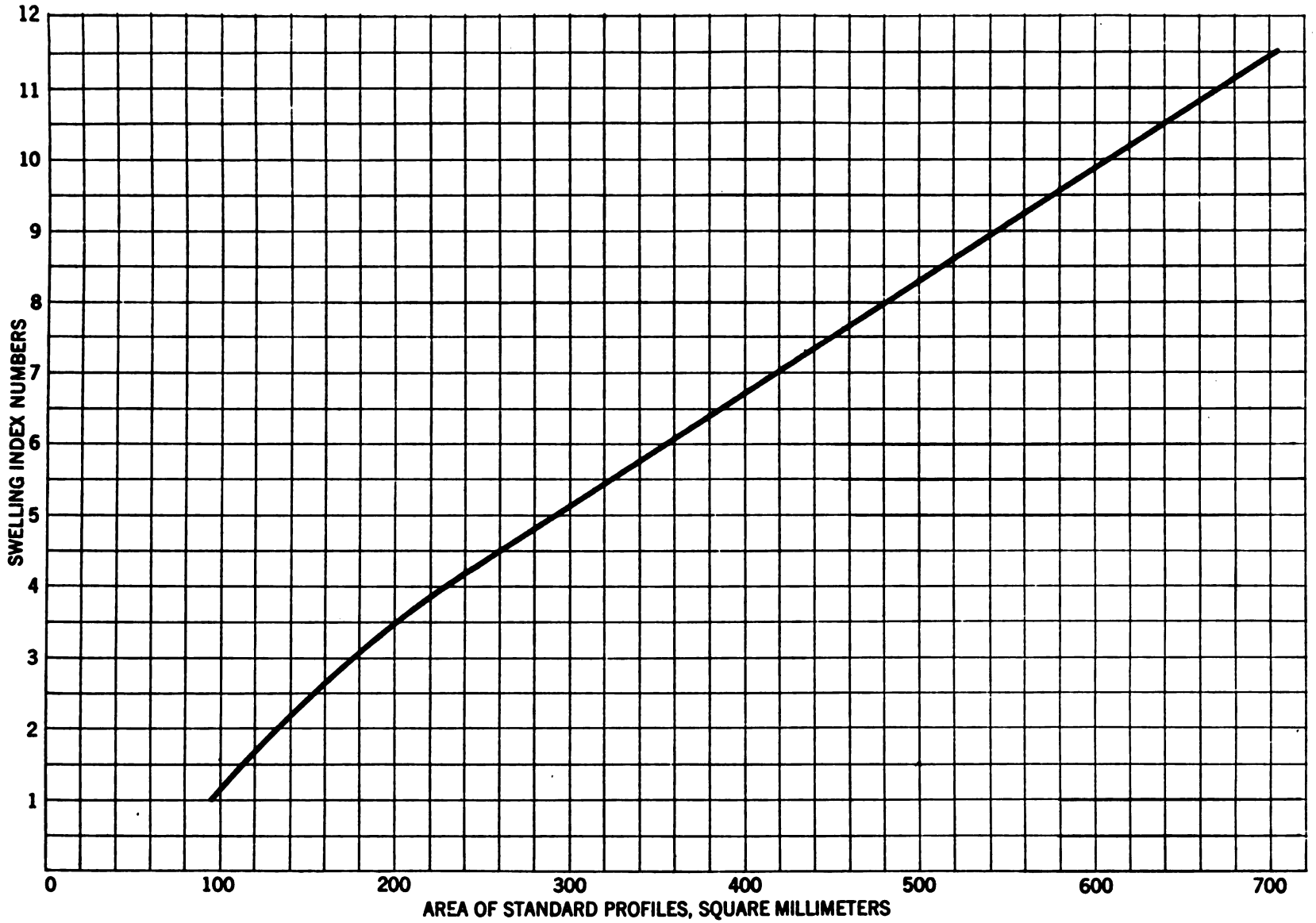

Figure 22. - Relationship of areas of standard profiles to swelling index number.

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Experiments made to determine whether there was any correlation between the results of the free-swelling test and expansion or contraction that takes place during carbonization of coal in coke ovens showed that no relationship existed. The "free-swelling" property of coal heated under no restraining influence must not be confused with the "expansion" property of coal when carbonized in a confining space.

Difficulty has been experienced in assigning swelling numbers to coals that give buttons that show no relationship in shape to the standard profiles. In addition, some buttons are larger than that of standard profile 9, the largest profile in the series. The use of some other method of measurement of the coke buttons makes the test somewhat more laborious, but it appears to be necessary for troublesome coals. The modifications that have been tried for evaluating the coke buttons of nonstandard shape were volume and cross-sectional area measurements. The latter method has proved to be more satisfactory. The scheme adopted was to determine the relationship of the areas of the standard profiles to the swelling index numbers. A curve based upon these data is shown in figure 22. When a coke button of unusual shape is obtained, its outline is drawn on paper and the area is measured. The area obtained can then be referred to the curve for determination of the corresponding swelling number.

Wartime Progress in Coke Production

There was a marked advance in the preparation of by-product coal due to changes in mining practice and developments in mechanical cleaning.[59/] However, the search for and development of new coals suitable for byproduct coking did not keep pace with preparation methods.

Competition in oven design has resulted in high efficiency in operation of the oven proper. The mechanical and electrical equipment used in the handling of coal and coke at the ovens has been improved considerably.

Few changes have been made in the basic design of the coke-handling plant, but there has been much progress in turning out a more uniform product through blending and control of the bulk density of the coal charge. Methods of testing coke are under investigation.

The financial returns from coke-oven byproducts may be increased by the recovery of new products such as pyridine and by efforts to produce more highly refined and pure aromatic hydrocarbons.

Survey of Carbonizing Properties of American Coals

Work on the Survey of Carbonizing Properties of American Coals was continued. This work included application of the test procedure developed by the Bureau of Mines in cooperation with the American Gas Association (known

59/ Seymour, William, Wartime Progress in Coke Production: Bureau of Mines Rept. of Investigations 3907, 1946, 13 pp.

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as the BM-AGA method), as well as studies of oxidation, plasticity, and expansion of coals. These methods have been used for the past 16 years in a Survey of the Gas,- Coke,- and Byproduct-Making Properties of American Coals and were designed to include investigation of many factors known to bear on the carbonizing properties of coal. During the war problems arose in the adaptation of new coals to the manufacture of metallurgical coke, and only that part of the procedure applying to high-temperature carbonization was used. Several of these special problems were incomplete at the close of the war, and the laboratory was occupied with rounding out such special investigations during most of the year.

Results of Carbonization Tests

The sources of coals investigated are given in table 10, and their analyses are given in table 11. Analyses of standard coals used for blending also are given.

Tables 12 to 15, inclusive, give the results of carbonization tests made by the BM-AGA procedure. Table 12 gives the yields of carbonization products; tables 13 and 13a, the physical properties of the coke; table 14, the properties of the gas; and table 15, the properties of tar distillates and light oils.

TABLE 10. - Sources of coals tested

Coal No.	Bed	Mine	Town	County	State
253	(Not named)	Vulcano	Punta Arenas	Province of Magallanes	Chile
254	do.	Elena	do.	do.	Do.
230	do.	Lirquen		Province of Concepcion	Do.
231	do.	Copihues de Pupunahue		Province of Valdivia	Do.
279					Peru
245		McAlester Strip	Rockdale	Milan	Texas
258	Powellton	Powellton No. 2	Ansted	Fayette	W. Va.
259	No. 2 Gas and Peerless	No. 3 North	do.	Fayette	Do.
90	Beckley	Stanaford	Mt. Hope	Raleigh	Do.
280	Pocahontas No. 4	No. 6	Eastgulf	do.	Do.
281	Pocahontas No. 3	No. 2 and 3	do.	do.	Do.
276	Lower Sunnyside	Sunnyside		Carbon	Utah
284			Coal Creek	Gunnison	Colorado
285			do.	do.	Do.
286			do.	do.	Do.

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TABLE 11. - Analyses of coals[1/]

Coal No.	Description	Dry, mineral-matter-free, fixed carbon, percent	Condition[2/]	Proximate, percent				Ultimate, percent					Air drying loss, percent	Heating value, B.t.u. per lb.	Softening temperature of ash, °F.
				Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur			
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
253	100 percent Vulcano mine	48.5	1	30.0	30.0	27.0	13.0	6.5	41.3	0.6	38.3	0.3	15.5	7,230	2,190
			2	-	42.9	38.6	18.5	4.5	59.0	.8	16.8	.4	-	10,340	-
			3	-	52.7	47.3	-	5.5	72.5	1.0	20.6	.4	-	12,690	-
254	100 percent Elena mine	50.8	1	21.0	33.7	33.6	11.7	6.2	49.7	.6	31.5	.3	13.1	8,760	2,530
			2	-	42.6	42.6	14.8	4.9	62.9	.8	16.2	.4	-	11,090	-
			3	-	50.1	49.9	-	5.8	73.9	.9	18.9	.5	-	13,020	-
230	100 percent Lirquen mine	51.5	1	15.1	35.7	36.5	12.7	5.9	56.3	1.0	23.4	.7	4.9	9,990	2,250
			2	-	42.1	43.0	14.9	4.9	66.3	1.2	11.8	.9	-	11,770	-
			3	-	49.4	50.6	-	5.8	77.9	1.4	13.9	1.0	-	13,830	-
231	100 percent Copihues de Pupunahue mine	56.2	1	20.7	31.7	39.6	8.0	6.0	52.9	1.1	31.7	.3	9.8	9,040	2,470
			2	-	40.0	49.9	10.1	4.6	66.7	1.4	16.9	.3	-	11,400	-
			3	-	44.5	55.5	-	5.2	74.3	1.5	18.6	.4	-	12,690	-
279	100 percent coal from Peru	78.8	1	.7	20.2	72.1	7.0	4.4	83.0	.9	3.7	1.0	0.3	14,410	2,700
			2	-	20.4	72.5	7.1	4.3	83.6	.9	3.1	1.0	-	14,520	-
			3	-	21.9	78.1	-	4.7	90.0	1.0	3.2	1.1	-	15,620	-
245	100 percent McAlester strip mine	50.0	1	24.2	33.4	32.3	10.1	6.3	48.2	.9	33.5	1.0	14.8	8,310	2,290
			2	-	44.0	42.6	13.4	4.7	63.5	1.2	15.9	1.3	-	10,960	-
			3	-	50.8	49.2	-	5.5	73.3	1.4	18.3	1.5	-	12,640	-
258	100 percent Powellton bed	62.1	1	1.6	34.9	55.9	7.6	5.4	77.6	1.5	7.1	.8	0.4	13,890	2,290.
			2	-	35.4	56.9	7.7	5.3	78.9	1.5	5.8	.8	-	14,120	-
			3	-	38.4	61.6	-	5.7	85.5	1.6	6.3	.9	-	15,290	-
259	100 percent No. 2 Gas and Peerless bed	63.3	1	1.8	33.6	56.3	8.3	5.2	77.0	1.5	7.1	.9	0.6	13,710	2,890
			2	-	34.3	57.2	8.5	5.1	78.5	1.5	5.5	.9	-	13,970	-
			3	-	37.4	62.6	-	5.6	85.7	1.6	6.1	1.0	-	15,270	-
75	100 percent Pocahontas No. 3 bed	81.8	1	2.4	17.2	74.5	5.9	4.5	83.0	1.1	4.9	.6	2.0	14,360	2,330
			2	-	17.6	76.3	6.1	4.3	85.0	1.2	2.8	.6	-	14,720	-
			3	-	18.7	81.3	-	4.6	90.6	1.3	2.8	.7	-	15,670	-
90	100 percent Beckley bed	79.5	1	2.3	19.7	74.8	3.2	4.8	85.3	1.6	4.5	.6	1.8	14,890	2,550
			2	-	20.2	76.6	3.2	4.7	87.4	1.6	2.4	.7	-	15,240	-
			3	-	20.8	79.2	-	4.9	90.3	1.7	2.4	.7	-	15,750	-
28	100 percent Pittsburgh bed	61.5	1	1.5	35.9	56.3	6.3	-	-	-	-	.9	0.5	14,010	2,780
			2	-	36.4	57.2	6.4	-	-	-	-	.9	-	14,230	-
			3	-	38.9	61.1	-	-	-	-	-	1.0	-	15,190	-
280	100 percent Pocahontas No. 4 bed	83.3	1	1.7	15.9	75.4	7.0	4.4	82.9	1.2	3.8	.7	1.1	14,350	2,850
			2	-	16.1	76.8	7.1	4.2	84.3	1.2	2.5	.7	-	14,600	-
			3	-	17.4	82.6	-	4.6	90.8	1.3	2.5	.8	-	15,720	-
281	100 percent Pocahontas No. 3 bed	83.3	1	1.7	15.9	75.7	6.7	4.3	82.9	1.1	4.2	.8	1.2	14,360	2,910+
			2	-	16.2	77.0	6.8	4.2	84.4	1.2	2.6	.8	-	14,610	-
			3	-	17.3	82.7	-	4.5	90.5	1.3	2.9	.8	-	15,670	-
276	100 percent Lower Sunnyside bed	57.0	1	3.7	38.9	50.5	6.9	5.5	73.3	1.6	11.2	1.5	1.4	13,170	2,550
			2	-	40.4	52.4	7.2	5.3	76.2	1.6	8.1	1.6	-	13,680	-
			3	-	43.6	56.4	-	5.7	82.0	1.8	8.8	1.7	-	14,730	-
284	100 percent core-drill hole No. 10-9, Paonia, Colo. coal log. 932 ft. 8-1/2-inch, 936 ft. 9-1/2-inch.	57.3	1	2.4	40.4	53.5	3.7	5.7	77.8	1.6	10.2	1.0	0.8	14,030	2,160
			2	-	41.3	54.9	3.8	5.5	79.7	1.7	8.2	1.1	-	14,370	-
			3	-	43.0	57.0	-	5.7	82.9	1.7	8.6	1.1	-	14,940	-
285	100 percent core-drill hole No. 10-9, Paonia, Colo. coal log. 946 ft. 1-inch, 952 ft. 7-inch.	57.1	1	2.4	39.9	52.3	5.4	5.6	76.9	1.5	10.1	.5	1.0	13,770	2,140
			2	-	40.9	53.5	5.6	5.5	78.8	1.5	8.1	.5	-	14,100	-
			3	-	43.3	56.7	-	5.8	83.4	1.6	8.7	.5	-	14,940	-
286	100 percent core-drill hole No. 10-9, Paonia, Colo. coal log. 958 ft. 2-inch, 964 ft. 11-inch.	58.2	1	2.1	38.4	52.4	7.1	5.5	75.7	1.5	9.7	.5	0.7	13,630	2,690
			2	-	39.3	53.4	7.3	5.4	77.3	1.5	8.0	.5	-	13,920	-
			3	-	42.4	57.6	-	5.8	83.4	1.7	8.5	.6	-	15,020	-

1/ Analyses by H. M. Cooper, chemist, Bureau of Mines, Pittsburgh, Pa.
2/ 1, Sample as received; 2, moisture-free; 3, moisture- and ash-free.

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TABLE 12. - Yields of carbonization products, as-carbonized basis

Coal 253 - 100 percent Vulcano mine
Coal 254 - 100 percent Elena mine
Coal 230 - 100 percent Lirquen mine
Coal 231 - 100 percent Copihues de Pupunahue mine
Coal 279 - 100 percent Peruvian coal
Coal 279A - 80 percent Lower Sunnyside bed and 20 percent Peruvian
Coal 258 - 100 percent Powellton bed
Coal 258A - 80 percent Powellton bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 258B - 70 percent Powellton bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 259 - 100 percent No. 2 Gas and Peerless bed
Coal 259A - 80 percent No. 2 Gas and Peerless bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 259B - 70 percent No. 2 Gas and Peerless bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 90 - 100 percent Beckley bed
Coal 90A - 80 percent Pittsburgh bed and 20 percent Beckley bed
Coal 90B - 70 percent Pittsburgh bed and 30 percent Beckley bed
Coal 280 - 100 percent Pocahontas No. 4 bed
Coal 281 - 100 percent Pocahontas No. 3 bed
Coal 276 - 100 percent Lower Sunnyside bed
Coal 276A - 90 percent Lower Sunnyside bed and 10 percent Lower Sunnyside char
Coal 276B - 90 percent Lower Sunnyside bed and 10 percent Gilsonite char
Coal 284 - Hole No. 10-9, Core Sample, Gunnison County, Colorado - 932'8-1/2" - 936'9-1/2"
Coal 285 - Hole No. 10-9, Core Sample, Gunnison County, Colorado - 946'1" - 952'7"
Coal 286 - Hole No. 10-9, Core Sample, Gunnison County, Colorado - 958'2" - 964'11"

Coal No.	Retort diameter, inches	Carbonizing temperature, °C.	Yields, percent by weight of coal[1]							Yields per ton of coal[1]				$(NH_4)_2SO_4$, pounds
			Coke	Gas	Tar	Light oil	Free ammonia	Liquor	Total	Gas, cu.ft.	Tar, gallons	Light oil, gal. In gas	Light oil, gal. To 170°C.	
253	10	1,000	41.2	30.7	0.9	0.55	0.032	21.9	95.3	16,350	1.8	1.54	0.04	8.0
253	13	600	45.6	14.2	3.1	.43	.024	33.1	96.5	5,650	7.5	1.28	.28	4.5
254	10	1,000	46.5	25.6	2.0	.65	.041	19.2	94.0	15,350	4.3	1.81	.10	8.5
254	13	600	51.3	14.2	4.5	.56	.009	26.5	97.1	5,650	10.9	1.69	.60	5.2
230	13	600	56.7	12.7	6.1	.63	.030	20.5	96.7	5,200	14.7	1.95	.85	14.0
231	13	600	53.4	11.8	2.4	.41	.014	28.9	96.9	4,750	5.6	1.23	.18	6.2
279	13	900	83.7	10.2	2.3	.43	.099	1.7	98.4	10,200	4.7	1.19	.08	8.7
279A	13	900	68.1	15.9	5.2	.78	.114	7.2	97.3	10,450	10.7	2.12	.68	20.3
258	18	900	70.1	14.6	6.9	1.11	.163	5.6	98.5	10,400	14.2	3.09	.65	17.7
258A	18	900	73.1	13.5	5.6	.96	.142	5.5	98.8	10,300	11.6	2.67	.60	18.5
258B	18	900	74.4	13.0	5.4	.85	.145	5.4	99.2	10,200	11.2	2.37	.50	18.1
259	18	900	71.3	14.2	6.3	1.17	.183	5.6	98.8	10,250	12.8	3.20	.54	19.9
259A	18	900	73.7	13.2	5.4	.97	.192	5.2	98.7	10,050	11.1	2.68	.45	20.4
259B	18	900	75.1	12.7	5.2	.82	.157	5.4	99.4	10,050	10.9	2.30	.59	19.7
90	13	600	84.1	7.4	3.6	.40	.059	4.3	99.9	4,450	7.9	1.18	.42	7.8
90	13	700	81.5	9.5	3.4	.47	.212	4.5	99.6	7,100	7.3	1.37	.32	22.7
90	13	800	80.7	10.3	3.3	.48	.219	3.8	98.8	9,350	6.7	1.33	.24	21.3
90[2]	13	800	81.0	10.6	3.0	.43	.205	4.1	99.3	9,150	6.3	1.20	.19	22.6
90[3]	13	800	80.9	10.7	2.2	.50	.193	4.3	98.8	9,300	4.6	1.38	.17	23.4
90[4]	13	800	80.6	10.9	1.9	.50	.165	5.5	99.6	9,400	4.0	1.39	.14	22.3
90	18	900	80.6	11.1	3.0	.59	.181	4.0	99.5	10,700	6.2	1.63	.15	19.4
90A	18	900	72.0	13.6	6.5	1.01	.136	5.5	98.7	10,350	13.6	2.84	1.02	21.5
90B	18	900	73.1	13.6	6.0	1.03	.152	5.4	99.3	10,450	12.5	2.88	.83	22.4
90	18	1,000	80.6	11.1	2.6	.54	.145	3.7	98.7	11,700	5.2	1.49	.09	14.5
280	13	800	84.3	9.4	1.9	.33	.165	3.4	99.5	8,900	3.8	.91	.09	15.5
280[5]	13	800	84.3	-	1.7	.33	-	3.5	-	-	3.4	.90	.07	-
280[6]	13	800	84.4	9.4	1.5	.32	0.155	3.6	99.4	8,600	3.0	.87	.05	15.7
280[7]	13	800	84.6	9.5	1.2	.31	.148	3.9	99.7	8,800	2.3	.84	.02	15.9
281	13	800	84.2	9.5	1.8	.29	.164	3.0	99.0	8,850	3.7	.80	.07	15.0
281[8]	13	800	84.4	9.6	1.8	.28	.169	3.3	99.5	8,750	3.7	.77	.06	18.1
281[9]	13	800	84.7	9.7	1.4	.36	.162	3.0	99.3	8,850	2.8	.99	.04	16.1
281[10]	13	800	84.7	9.3	1.1	.26	.181	2.9	98.4	8,800	2.3	.70	.03	17.7
276	13	900	64.1	17.0	6.4	.93	.101	8.6	97.1	10,550	13.2	2.54	1.00	16.4
276A	13	900	66.4	16.2	5.8	.85	.116	8.6	98.0	10,350	11.9	2.32	.81	23.7
276B	13	900	66.6	16.1	5.9	.89	.134	8.0	97.6	10,400	12.1	2.44	.84	23.9
284	13	900	64.2	17.4	7.5	.85	.124	7.7	97.8	10,800	15.7	2.35	1.13	21.7
285	13	900	64.5	18.1	6.7	.95	.128	7.0	97.4	11,100	13.8	2.62	.80	20.4
286	13	900	65.2	17.3	7.2	.87	.120	7.3	98.0	10,500	15.0	2.43	1.11	20.9

1/ Coke, tar, ammonia, and light oil are reported moisture-free; gas is reported as stripped of light oil and saturated with water vapor at 60° F. and under a pressure of 30 inches of mercury.
Time oxidized in air at 100° C., days: 2/, 2.8; 3/, 10.2; 4/, 18.4; 5/, 3.8; 6/, 9.0; 7/, 14.2; 8/, 5.0; 9/, 11.0; 10/, 13.1.

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TABLE 13. - Physical properties of coke

Coal 258 - 100 percent Powellton bed
Coal 258A - 80 percent Powellton bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 258B - 70 percent Powellton bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 259 - 100 percent No. 2 Gas and Peerless bed
Coal 259A - 80 percent No. 2 Gas and Peerless bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 259B - 70 percent No. 2 Gas and Peerless bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 90 - 100 percent Beckley bed
Coal 90A - 80 percent Pittsburgh bed and 20 percent Beckley bed
Coal 90B - 70 percent Pittsburgh bed and 20 percent Beckley bed
Coal 280 - 100 percent Pocahontas No. 4 bed
Coal 281 - 100 percent Pocahontas No. 3 bed

Coal No.	Retort diameter, inches	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon-				Tumbler test, cumulative percent upon-			
						2-in. screen	1-1/2-in. screen	1-in. screen	1/4-in. screen	2-in. screen	1-1/2-in. screen	1-in. screen	1/4-in. screen
258	18	900	1.90	0.89	53.2	54.5	82.5	94.0	98.0	1.8	28.5	53.9	67.2
258A	18	900	1.91	.88	53.9	58.2	85.8	95.7	98.5	4.8	33.0	59.1	70.5
258B	18	900	1.91	.88	53.9	55.6	84.2	95.2	98.6	6.6	35.9	60.9	72.0
259	18	900	1.89	.91	51.9	54.8	80.6	93.9	98.3	2.9	31.9	58.5	70.8
259A	18	900	1.90	.92	51.6	49.9	81.2	94.8	98.6	4.9	30.9	61.3	74.8
259B	18	900	1.91	.90	52.9	53.2	80.8	95.0	98.7	5.0	32.5	59.9	73.7
90	13	600	1.57	.75	52.2	92.8	97.1	97.7	98.6	41.0	62.5	65.1	65.4
90	13	700	1.72	.81	52.9	94.5	96.9	98.1	98.8	45.0	65.7	68.2	68.9
90	13	800	1.85	.82	55.7	80.6	94.4	97.4	98.7	26.9	58.3	66.6	68.6
90[1/]	13	800	1.84	.83	54.9	86.9	96.2	98.2	99.0	29.4	59.5	68.7	69.5
90[2/]	13	800	1.83	.83	54.6	85.6	94.6	97.3	98.8	33.3	55.9	63.7	65.7
90[3/]	13	800	1.85	-	-	-	-	-	-	1.3	18.4	32.9	39.4
90	18	900	1.89	0.86	54.5	60.1	86.0	96.3	98.8	14.8	43.1	66.0	72.2
90A	18	900	1.89	.81	57.1	44.0	79.8	94.6	98.5	4.3	30.1	62.3	72.6
90B	18	900	1.89	.85	55.0	46.1	80.9	95.2	98.5	6.6	32.1	63.2	73.7
90	18	1,000	1.93	.87	54.9	24.9	67.0	94.5	99.0	.4	19.3	60.7	75.5
280	13	800	-	.88	-	80.1	93.1	96.7	98.7	18.5	44.9	57.7	62.1
280[4/]	13	800	-	.85	-	79.9	88.9	94.8	97.3	15.4	40.0	52.5	56.2
280[5/]	13	800	-	.86	-	-	-	-	-	5.0	24.8	40.2	44.6
281	13	800	-	.87	-	82.8	94.2	97.4	98.6	14.8	50.6	61.1	63.9
281[6/]	13	800	-	.84	-	80.3	94.0	95.9	97.9	15.1	46.4	53.9	55.9

Time oxidized in air at 100° C., days: 1/, 2.8; 2/, 10.2; 3/, 18.4; 4/, 3.8; 5/, 9.0; 6/, 5.0.

TABLE 13a. - Physical properties of coke (Columbia Steel Co. methods)

Coal 279 - 100 percent Peruvian coal
Coal 279A - 80 percent Lower Sunnyside bed (coal 276) and 20 percent Peruvian
Coal 284 - Hole No. 10-9, core sample, Gunnison County, Colorado - 932'8-1/2" - 936'9-1/2"
Coal 285 - Hole No. 10-9, core sample, Gunnison County, Colorado - 946'1" - 952'7"
Coal 286 - Hole No. 10-9, core sample, Gunnison County, Colorado - 958'2" - 964'11"
Coal 276 - 100 percent Lower Sunnyside bed
Coal 276A - 90 percent Lower Sunnyside bed and 10 percent Lower Sunnyside char
Coal 276B - 90 percent Lower Sunnyside bed and 10 percent Gilsonite char

Coal No.	Retort diameter, inches	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon-				Tumbler test, cumulative percent upon-			
						1-1/2-in. screen	1-in. screen	3/4-in. screen	1/2-in. screen	1-1/2-in. screen	1-in. screen	3/4-in. screen	1/2-in. screen
279	13	900	1.92	0.87	54.7	68	94	97	98	47	79	83	86
279A	13	900	1.93	.81	58.0	58	90	95	98	36	75	82	86
284	13	900	1.90	.73	61.6	51	81	88	94	22	62	71	80
285	13	900	1.90	.73	61.6	28	70	80	91	19	53	64	76
286	13	900	1.92	.76	60.4	37	80	89	96	25	65	76	84
276	13	900	1.93	.75	61.1	43	75	84	92	20	55	65	76
276A	13	900	1.94	.82	57.7	62	88	93	96	27	67	75	82
276B	13	900	1.93	.81	58.0	64	89	93	96	30	67	73	78

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TABLE 14. - Physical and chemical properties of gas

Coal 253 - 100 percent Vulcano mine
Coal 254 - 100 percent Elena mine
Coal 230 - 100 percent Lirquen mine
Coal 231 - 100 percent Copihues de Pupunahue mine
Coal 279 - 100 percent Peruvian coal
Coal 279A - 80 percent Lower Sunnyside bed and 20 percent Peruvian
Coal 258 - 100 percent Powellton bed
Coal 258A - 80 percent Powellton bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 258B - 70 percent Powellton bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 259 - 100 percent No. 2 Gas and Peerless bed
Coal 259A - 80 percent No. 2 Gas and Peerless bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 259B - 70 percent No. 2 Gas and Peerless bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 90 - 100 percent Beckley bed
Coal 90A - 80 percent Pittsburgh bed and 20 percent Beckley bed
Coal 90B - 70 percent Pittsburgh bed and 30 percent Beckley bed
Coal 280 - 100 percent Pocahontas No. 4 bed
Coal 281 - 100 percent Pocahontas No. 3 bed
Coal 276 - 100 percent Lower Sunnyside bed
Coal 276A - 90 percent Lower Sunnyside bed and 10 percent Lower Sunnyside char
Coal 276B - 90 percent Lower Sunnyside bed and 10 percent Gilsonite char
Coal 284 - Hole No. 10-9, core sample, Gunnison County, Colorado - 932'8-1/2" - 936'9-1/2"
Coal 285 - Hole No. 10-9, core sample, Gunnison County, Colorado - 946'1" - 952'7"
Coal 286 - Hole No. 10-9, core sample, Gunnison County, Colorado - 958'2" - 964'11"

Coal No.	Retort diameter, inches	Carbonizing temperature, °C.	Specific gravity	Gross heating value[1] B.t.u. per cubic foot Determined	Gross heating value[1] B.t.u. per cubic foot Calculated	Gross heating value[1] B.t.u. per pound of coal	H_2S, grains per 100 cubic feet	Composition, dry, percent by volume CO_2	Illuminants	O_2	H_2	CO	CH_4	C_2H_6	N_2
253	10	1,000	0.497	365	379	2,980	70	9.1	2.1	0.4	53.8	24.7	7.1	1.5	1.3
253	13	600	.667	499	471	1,410	200	24.9	2.3	.3	38.5	6.0	24.0	3.1	.9
254	10	1,000	.444	456	410	3,500	80	8.8	3.3	.4	52.4	21.9	11.9	.2	1.1
254	13	600	.668	543	519	1,530	200	22.2	2.9	.3	36.6	5.9	27.5	3.7	.9
230	13	600	.644	633	602	1,650	440	17.8	4.0	.3	34.1	6.0	30.2	6.4	1.2
231	13	600	.663	561	559	1,330	180	22.1	2.2	.3	30.2	7.6	31.4	5.3	.9
279	13	900	.264	522	509	2,660	310	1.0	1.7	.4	66.1	3.2	26.4	.0	1.2
279A	13	900	.403	605	587	3,160	580	2.5	5.0	.3	50.7	8.2	30.6	1.0	1.7
258	18	900	.371	618	606	3,210	240	1.9	4.9	.2	52.8	5.5	32.0	1.5	1.2
258A	18	900	.347	592	578	3,050	200	1.5	4.0	.2	56.3	4.7	31.3	.6	1.4
258B	18	900	.337	585	570	2,980	190	1.4	3.4	.3	57.9	4.5	30.2	1.1	1.2
259	18	900	.367	605	593	3,100	390	1.8	4.8	.2	53.7	5.7	31.8	.8	1.2
259A	18	900	.349	590	579	2,960	330	1.8	4.1	.3	56.2	5.1	30.8	.8	.9
259B	18	900	.335	578	570	2,900	300	1.5	3.6	.4	58.2	4.7	28.9	1.5	1.2
90	13	600	.440	747	736	1,660	190	1.3	1.0	.4	38.3	2.3	50.6	5.1	1.0
90	13	700	.356	641	636	2,280	190	.9	1.5	.4	50.8	2.6	40.1	2.6	1.1
90	13	800	.292	561	554	2,620	170	.8	2.2	.4	61.9	2.7	29.7	1.1	1.2
90[2]	13	800	.306	568	562	2,600	160	.8	2.0	.4	60.8	2.7	30.8	1.3	1.2
90[3]	13	800	.307	562	553	2,610	160	1.4	2.1	.4	60.1	3.7	30.2	1.0	1.1
90[4]	13	800	.308	-	534	-	130	1.7	2.2	.4	61.3	4.0	28.9	.3	1.2
90	18	900	.275	533	528	2,850	150	.6	2.0	.4	65.0	2.5	27.9	.3	1.3
90A	18	900	.349	592	583	3,060	310	1.4	3.7	.4	55.3	5.5	31.3	1.2	1.2
90B	18	900	.346	587	579	3,070	300	1.3	3.4	.3	56.4	5.1	30.8	1.4	1.3
90	18	1,000	.252	517	500	3,020	160	.6	1.6	.4	68.2	3.1	24.9	.0	1.2
280[5]	13	800	.280	529	525	2,350	210	.9	1.6	.4	64.2	3.6	27.6	.6	1.1
280[6]	13	800	0.292	528	525	2,270	210	1.0	1.7	0.1	63.4	3.3	28.6	0.1	1.8
280[7]	13	800	.287	515	515	2,270	190	1.5	1.5	.4	64.5	3.3	27.8	.0	1.0
281	13	800	.286	533	531	2,360	180	.7	1.5	.3	63.7	2.9	29.0	.5	1.4
281[8]	13	800	.292	529	-	2,310	180	-	-	-	-	-	-	-	-
281[9]	13	800	.289	513	508	2,270	180	1.4	1.4	0.4	64.6	3.5	27.1	.0	1.6
281[10]	13	800	.281	520	515	2,290	170	1.6	1.7	.4	64.3	3.4	27.4	.0	1.2
276	13	900	.428	625	609	3,300	610	3.2	6.4	.3	47.2	9.3	31.4	1.0	1.2
276A	13	900	.416	605	589	3,130	560	3.2	5.4	.2	49.6	9.1	30.0	1.2	1.3
276B	13	900	.413	607	588	3,160	550	3.2	5.9	.2	49.9	8.5	30.2	.6	1.5
284	13	900	.429	651	629	3,520	400	2.2	6.6	.3	46.8	7.7	34.5	.6	1.3
285	13	900	.432	652	630	3,620	170	2.6	6.7	.4	45.6	8.8	33.9	.9	1.1
286	13	900	.438	656	631	3,440	190	2.4	7.0	.4	45.8	7.5	35.0	.3	1.6

1/ Stripped of light oil and saturated with water vapor at 60° F. and under a pressure of 30 inches of mercury. Time oxidized in air at 100° C., days: 2/, 2.8; 3/, 10.2; 4/, 18.4; 5/, 3.8; 6/, 9.0; 7/, 14.2; 8/, 5.0; 9/, 11.0; 10/, 13.1.

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TABLE 15. - Analysis of tar distillate and light oil

Coal 253 - 100 percent Vulcano mine
Coal 254 - 100 percent Elena mine
Coal 230 - 100 percent Lirquen mine
Coal 231 - 100 percent Copihues de Pupunahue mine
Coal 279 - 100 percent Peruvian coal
Coal 279A - 80 percent Lower Sunnyside bed and 20 percent Peruvian
Coal 258 - 100 percent Powellton bed
Coal 258A - 80 percent Powellton bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 258B - 70 percent Powellton bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 259 - 100 percent No. 2 Gas and Peerless bed
Coal 259A - 80 percent No. 2 Gas and Peerless bed and 20 percent Pocahontas No. 3 bed (coal 75)
Coal 259B - 70 percent No. 2 Gas and Peerless bed and 30 percent Pocahontas No. 3 bed (coal 75)
Coal 90 - 100 percent Beckley bed
Coal 90A - 80 percent Pittsburgh bed and 20 percent Beckley bed
Coal 90B - 70 percent Pittsburgh bed and 30 percent Beckley bed
Coal 280 - 100 percent Pocahontas No. 4 bed
Coal 281 - 100 percent Pocahontas No. 3 bed
Coal 276 - 100 percent Lower Sunnyside bed
Coal 276A - 90 percent Lower Sunnyside bed and 10 percent Lower Sunnyside char
Coal 276B - 90 percent Lower Sunnyside bed and 10 percent Gilsonite char
Coal 284 - Hole No. 10-9, core sample, Gunnison County, Colorado - 932'8-1/2" - 936'9-1/2"
Coal 285 - Hole No. 10-9, core sample, Gunnison County, Colorado - 946'1" - 952'7"
Coal 286 - Hole No. 10-9, core sample, Gunnison County, Colorado - 958'2" - 964'11"

Coal No.	Retort diameter, inches	Carbonizing temperature, °C.	Distillate, percent by volume of dry tar			Neutral tar oil, percent by volume			Refined light oil from gas, percent by volume				Olefins in crude light oil from gas, percent by volume
			Acids	Bases	Neutral oils	Olefins	Aromatics	Paraffins and naphthenes	Benzene	Toluene	Paraffins	Solvent naphtha	
253	10	1,000	8.4	1.9	38.2	14.1	76.9	9.0	46.5	22.6	6.0	24.9	21.7
253	13	600	15.4	2.4	49.3	16.8	58.0	25.2	25.1	21.3	28.7	24.9	36.1
254	10	1,000	9.8	1.8	36.5	17.0	74.3	8.7	60.6	21.2	5.1	13.1	20.9
254	13	600	19.9	1.8	32.2	14.0	58.5	27.5	20.1	19.7	30.7	29.5	37.1
230	13	600	16.9	2.5	45.9	16.0	48.9	35.1	18.7	16.6	38.1	26.6	43.2
231	13	600	23.5	2.4	44.3	15.0	55.7	29.3	23.4	21.4	30.1	25.1	34.2
279	13	900	1.0	.8	17.7	8.7	91.3	.0	63.4	26.8	1.6	8.2	10.3
279A	13	900	3.6	2.5	29.2	11.2	88.4	.4	72.4	22.7	1.4	3.5	10.9
258	18	900	5.6	1.9	27.2	10.5	85.5	4.0	65.7	20.4	5.8	8.1	13.1
258A	18	900	6.7	1.9	28.8	11.0	85.4	3.6	67.6	19.9	6.0	6.5	12.5
258B	18	900	8.2	2.1	31.1	11.3	82.4	6.3	60.2	21.7	8.1	10.0	13.4
259	18	900	4.0	1.7	23.5	9.5	89.2	1.3	71.9	20.2	3.6	4.3	10.1
259A	18	900	6.4	1.8	26.4	10.5	87.7	1.8	65.9	22.7	4.8	6.6	11.1
259B	18	900	8.9	2.1	32.4	9.8	84.5	5.7	59.5	21.3	8.6	10.6	15.2
90	13	600	6.5	.6	33.9	6.9	70.2	22.9	23.7	21.2	36.4	18.7	24.9
90	13	700	5.9	.8	30.0	6.8	78.7	14.5	20.1	29.7	21.3	28.9	21.4
90	13	800	3.7	1.1	24.2	9.0	84.0	7.0	36.9	33.6	9.4	20.1	16.9
90[1]	13	800	3.5	1.1	24.3	6.1	91.3	2.6	31.3	36.1	9.3	23.3	13.6
90[2]	13	800	2.4	1.8	23.7	5.8	93.3	.9	49.1	33.0	3.9	14.0	12.0
90[3]	13	800	3.9	1.8	24.0	8.1	90.7	1.2	46.9	34.2	4.3	14.6	12.9
90	18	900	2.6	.7	19.4	8.0	86.5	5.5	60.4	24.5	3.8	11.3	12.3
90A	18	900	7.4	1.9	32.0	11.0	81.9	7.1	63.4	18.8	8.9	8.9	13.2
90B	18	900	6.4	1.7	28.9	8.3	86.2	5.5	68.5	17.7	7.3	6.5	12.4
90	18	1,000	1.8	.8	18.0	8.3	90.3	1.4	70.2	19.1	2.4	8.3	9.8
280	13	800	1.8	.5	19.1	6.7	92.8	.5	45.4	37.8	2.9	13.9	11.3
280[4]	13	800	1.8	.9	19.6	6.6	92.5	.9	40.1	40.1	5.7	14.1	11.3
280[5]	13	800	2.0	.7	19.9	5.0	94.5	.5	34.0	41.9	6.6	17.5	12.9
280[6]	13	800	1.6	1.1	18.0	8.3	91.4	.3	43.4	38.4	4.1	14.1	12.8
281	13	800	2.4	1.1	18.3	9.1	90.5	.4	37.8	39.9	7.9	14.4	11.9
281[7]	13	800	2.4	.6	19.5	6.8	92.9	.3	37.0	42.2	4.0	16.8	10.6
281[8]	13	800	2.0	.9	18.1	7.6	91.9	.5	41.6	36.2	8.5	13.7	13.0
281[9]	13	800	2.0	1.0	18.9	6.6	92.9	.5	35.4	41.3	6.6	16.7	11.7
276	13	900	4.5	2.2	31.1	10.5	87.4	2.1	74.0	20.0	2.7	3.3	12.6
276A	13	900	4.8	2.1	30.6	12.5	85.6	1.9	73.4	20.0	2.9	3.7	12.3
276B	13	900	4.3	2.2	30.1	11.3	86.6	2.1	73.9	20.1	2.7	3.3	11.5
284	13	900	7.0	2.5	34.6	11.5	85.6	2.9	66.3	22.4	5.1	6.2	15.4
285	13	900	5.0	2.5	29.8	13.0	84.4	2.6	72.5	19.5	3.6	4.4	11.9
286	13	900	7.3	2.5	34.9	10.0	87.0	3.0	65.8	21.2	6.5	6.5	13.0

Time oxidized in air at 100° C., days: 1/, 3.9; 2/, 5.0; 3/, 7.2; 4/, 2.7; 5/, 7.5; 6/, 11.9; 7/, 3.7; 8/, 8.3; 9/, 11.5.

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230	13	600	56.7	12.7	6.1	.63	.030	20.5	96.7	5,200	14.7	1.95		.85		14.0		
231	13	600	53.4	11.8	2.4	.41	.014	28.9	96.9	4,750	5.6	1.23		.18		6.2		
254	10	1,000	.444		456		410	3,500		80	8.8	3.3	.4	52.4	21.9	11.9	.2	1.1
254	13	600	.668		543		519	1,530		200	22.2	2.9	.3	36.6	5.9	27.5	3.7	.9
230	13	600	.644		633		602	1,650		440	17.8	4.0	.3	34.1	6.0	30.2	6.4	1.2
231	13	600	.663		561		559	1,330		180	22.1	2.2	.3	30.2	7.6	31.4	5.3	.9
270	13	900	.264		522		509	2,660		310	1.0	1.7	.4	66.1	3.2	26.4	.0	1.2

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Normally, coals of high-volatile A rank or higher give material balances (table 12) totaling 98.5 to 99.5 percent, and this figure serves as a check on the accuracy of the work. However, with high-oxygen coals containing high percentages of water, such as the Chilean subbituminous coals, there is a loss of moisture, and the material balances may fall as low as 94 percent. A large proportion of the water at high carbonizing temperatures is used in the formation of water gas, which complicates the process. The weight of water so used should appear as oxides of carbon and hydrogen, but determination of yields of gas on a weight basis is subject to considerable error and, furthermore, it is possible that with excessive amounts of water in the gas it is difficult to determine the saturation at the point of measurement. The yields of ammonia at carbonizing temperatures above 800° C. are generally lower than those obtained in refractory ovens, because at high temperatures the iron walls of the BM-AGA retorts decompose ammonia much more rapidly than do refractories.

For determining the shatter and tumbler indexes of coke (table 13) two methods are used - the BM-AGA methods following those approved by the American Society for Testing Materials and by the Columbia Steel Co. The former applies to Appalachian and Midwestern coals and the latter to the less strongly coking coals of the West. For guidance in interpreting results for a given coal, average results are available for coals previously tested.

For example, an average high-volatile A coal when carbonized at 900° C., in the 18-inch retort will yield coke having a 1-1/2-inch shatter index of 70, a 1-inch tumbler index of 51, and a 1/4-inch tumbler index of 72. Higher indexes indicate, better than average coke-making properties, whether the coal is of high-volatile A rank. Low- and medium-volatile coals and their blends will yield stronger cokes with higher indexes. The index which the Columbia Steel Co. emphasize most in interpreting results by their method of test is the proportion remaining on the 1/2-inch screen in the tumbler test; this figure should be 75 or higher. The Beckley coal for which results by the Bureau of Mines method are given in the table 13 is of low-volatile rank; the quality of coke is about average for low-volatile coals. The average 1-1/2-inch shatter index is 85, and the average tumbler index is 64. The effect of mild oxidation of the coal upon the quality of the coke is indicated by the shatter and tumbler indexes for coals 280 and 281. Here, the effect upon the tumbler index is most pronounced. The tumbler test emphasizes abradability in the coke, and it appears that the main effect of oxidation upon quality of the coke is to render it more abradable.

Properties of gases from coals tested during the year are given in table 14. For the most part the gases are normal, considering the rank and grade of the coals. However, the high yield of gas from the subbituminous Elena coal (254) at 1,000° C. is abnormal; 3,500 B.t.u. in gas per pound of coal carbonized is as high as would be obtained from a good Appalachian gas coal; its heating value in B.t.u. per cubic foot, although not as high as would be obtained from a gas coal, is considered high enough for city distribution. The hydrogen sulfide content of this gas is very low - a desirable quality from the gas-maker's point of view.

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Table 15 gives the properties of the tar distillates and light oils. The subbituminous Chilean coals show high percentages of tar acids and low aromatic content as compared with other coals of the table. Also, the benzene content of the light oils is low and the olefin content is high. This composition is characteristic of bituminous coals of low rank. The composition of tars from other coals in the table is also characteristic of strongly coking coals, the class to which they belong.

Chilean Coals

The Chilean coals, Nos. 230, 231, 253, and 254,[60] were all subbituminous coals and noncoking. They were tested at the request of the Chilean Government. A "Technical Report of the Coal Mission on Coals of Chile," which included "Carbonizing Properties of Chilean Bituminous and Subbituminous Coals sampled by Engineers of the U. S. Bureau of Mines" and "Washing Characteristics of the Chilean Coals," was completed and transmitted through the Secretary of the Interior to the Secretary of State for transmittal to the Chilean Government. The main points of interest were yields of carbonization products at low temperatures and yield and quality of gases at high temperatures. Chile has no true gas coals and, accordingly, it was important to investigate all possible sources of gas for city distribution. The investigation showed that the Elena mine coal yielded 3,500 B.t.u. in gas per pound of coal carbonized, and the heating value per cubic foot was 456 B.t.u. It is considered feasible to supply such gas for domestic use. The low-temperature yields from these coals were as expected from coals of their rank.

Texas Lignite

Texas lignite (coal 245) mixed with asphaltic oil residues was carbonized to determine whether a coke of metallurgical quality could be produced from such mixtures. It is known that asphaltic oils possess considerable binding power, and cokelike residues are obtained when they are carbonized with totally inert carbonaceous matter. The experimental procedure consisted in preparation of the raw lignite by drying and also precarbonization, mixing with 10 to 30 percent of the oil residue, and carbonizing the mixture with and without briquetting. Raw lignite mixed with oil also was carbonized, but a very porous coked residue bearing little resemblance to true coke was obtained. It was finally found necessary to prepare the lignite by carbonization, mix it with a viscous oil residue (15 to 20 percent), make a briquette, and then carbonize at a high temperature. Coke briquets so produced were dense and hard, but they contained too much ash (about 20 percent); furthermore, it is doubtful whether a coke produced by such an expensive process would compete with that made from Appalachian region coals.

Peruvian Coal

A sample of Peruvian low-volatile coal taken by Thomas Fraser of the Bureau of Mines was carbonized at 900° C. A carbonization test was made,

60/ This series of numbers applies only to coals on which the complete BM-AGA procedure has not been used.

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also, in which 20 percent of this coal was blended with 80 percent Sunnyside coal from Carbon County, Utah. Sunnyside coal was chosen for blending because its coking quality is similar to that of Chilean coals, and it is possible that this Peruvian coal will be exported to Chile for blending purposes. The Chilean Government proposes to establish a small steel industry, and low-volatile coal is needed for blending with native coals to produce metallurgical coke.

The chemical composition of the Peruvian coal is given in table 11. It contained 20.2 percent volatile matter, the ash content was moderate (7.0 percent), and the sulfur was satisfactorily low (1.0 percent). The coke was fine-grained and contained few fractures. Table 13a shows its physical properties (279) and those of the blend (279A) with Sunnyside coal, the coke tests being made by the Columbia Steel methods. Coke from 100 percent Sunnyside coal is included for comparison. The enormous improvement in coke from Sunnyside coal by blending with the Peruvian coal is evident. Therefore, the latter must be considered a very satisfactory coal for blending with coals of Sunnyside rank.

Powellton and No. 2 Gas-Peerless Coals

Two West Virginia high-volatile coals that had been cleaned by a heavy-media (magnetite) process and their blends with 20 and 30 percent low-volatile coal were carbonized by the BM-AGA method at 900° C. Both coals were from Ansted, Fayette County, W. Va. Powellton coal was from the Powellton No. 2 mine, and No. 2 Gas-Peerless coal was from the No. 3 North mine. They were of similar rank and chemical composition (table 11); both contained moderate proportions of moisture, ash, and sulfur. The ash from Powellton softened at 2,290° F., whereas that from No. 2 Gas-Peerless, softened at 2,890° F. Their plastic properties also were similar - both coals attained high fluidity in the Gieseler test.

The yields in the BM-AGA test at 900° C. from Powellton and No. 2 Gas-Peerless coals were (table 12), respectively: Coke, 70.1 and 71.3 percent; and, upon the basis of per ton of coal, gas, 10,400 and 10,250 cubic feet; tar, 14.2 and 12.8 gallons; light oil in gas, 3.09 and 3.20 gallons; and ammonium sulfate, 17.7 and 19.9 pounds. Powellton coke resisted breakage in the shatter test more than No. 2 Gas-Peerless coke, but the latter was less abradable in the tumbler test. The 1-1/2-inch shatter and 1-inch tumbler indexes of both cokes were appreciably higher than the averages for 25 high-volatile A coals tested previously;[61] the averages for 25 coals were 70.4 and 50.8 for the 1-1/2-inch shatter and 1-inch tumbler, respectively. However, both 1/4-inch tumbler indexes were lower than the average 72.2. Blending with Pocahontas No. 3 coal strengthened the cokes from both coals. The abradability of Powellton coke was decreased significantly by blending with 20 percent of this low-volatile coal, but other effects were less

61/ Reynolds, D. A., and Holmes, C. R., Physical Properties of Cokes from Bureau of Mines-American Gas Association Tests at 800° and 900° C.: Bureau of Mines Rept. of Investigations 3650, 1942, 14 pp.

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pronounced. Blending with 30 percent low-volatile coal effected some further decrease in the abradability of Powellton coke; otherwise, the increase in the proportion of low-volatile did not improve the cokes from either coal. The gas from Powellton coal was slightly richer than that from No. 2 Gas-Peerless coal; the heating values per pound of coal were 3,210 and 3,100 B.t.u., respectively. The properties of the tars and light oils were normal for high-volatile A coals.

Beckley Coal

Beckley-bed coal from the Stanaford mine, Mt. Hope, Raleigh County, W. Va., contained 79.5 percent fixed carbon on the dry, mineral-matter-free basis and therefore ranks as a low-volatile coal. It was carbonized in 13-inch BM-AGA retorts at 600°, 700°, and 800° C. and in 18-inch retorts at 900° and 1,000° C. Oxidized samples were carbonized at 800° C., and blends with Pittsburgh-bed coal were carbonized at 900° C. Yields and properties of the carbonization products from these tests are given in tables 12, 13, 14, and 15.

This Beckley coal yielded 80.6 percent coke; 10,700 cubic feet of gas; 6.2 gallons of tar; 1.78 gallons of light oil; and 19.4 pounds of ammonium sulfate per ton at 900° C. The average yields for 11 low-volatile coals[62] differed appreciably from these in two respects - the average coke yield was 82.8 percent, and the average tar yield was 3.8 gallons per ton. The 900° C. coke was fine-grained and well-fused; its 1-1/2-inch shatter index was 86.0 and its 1- and 1/4-inch tumbler indexes were 66.0 and 72.2, respectively. The cokes made at lower temperatures had higher shatter indexes and lower 1/4-inch tumbler indexes. The 900° C. gas had heating values of 533 B.t.u. per cubic foot and 2,850 B.t.u. per pound of coal and contained 150 grains of hydrogen sulfide per cubic feet.

Blending Beckley coal with 70 and 80 percent Pittsburgh-bed coal lowered the yield of coke 7.5 to 8.6 percent and increased the percentage yields of other products except free ammonia. The yield of gas calculated to the volume basis decreased. The strength-test indexes of the cokes from the blends were very similar to the averages for the cokes from 10 low-volatile coal blends in which the same Pittsburgh-bed coal was used.[63] For example, the indexes for the 20 percent low-volatile blends were for Beckley and the average low-volatile coals, respectively: 1-1/2-inch shatter, 79.8 and 79.9; 1-inch tumbler, 62.3 and 61.5; and 1/4-inch tumbler, 72.6 and 72.4. The blend containing 30 percent Beckley coal yielded slightly stronger coke. Blending raised the specific gravity and heating value of the gas, these effects being due to a reduction in the hydrogen content.

In the investigation of Beckley coal the study of its expanding properties was emphasized. The carbonizing sample, blends with 70 and 80 percent

62/ Reynolds, D. A., and Davis, J. D., Blending Properties of Low- and Medium-Volatile Coals as Determined in the BM-AGA Apparatus: Bureau of Mines Rept. of Investigations 3936, 1946, 20 pp.

63/ See footnote 62.

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Pittsburgh coal; and six samples representing different sections of the mine were tested in the sole-heated oven; the blends were also tested in the vertical-slot oven. The carbonization sample expanded 15.9 percent in the sole-heated oven at a charge density of 55.5 pounds per cubic foot, which was more than the six mine samples expanded (9.0 to 13.6 percent; table 20). Pocahontas No. 3 coal from Kimball, McDowell County, W. Va., expanded 24.4 percent under similar test conditions.[64] The blend of 20 percent Beckley and 80 percent Pittsburgh coals contracted 3.6 percent in the sole-heated oven and developed a maximum pressure of 0.8 pound per square inch in the vertical-slot oven; the corresponding results for 30:70 blend were 1.0 percent expansion and pressure of 3.4 pounds per square inch. These blends probably could be carbonized safely in byproduct ovens; however, the 30:70 blend should not be charged at high densities, because it developed rather high pressure in the vertical oven.

Technical Paper 683[65] gives results of an investigation of the composition and properties of Powellton coal from Coal Mountain mine, Guyan, W. Va. This coal ranks as high-volatile A bituminous and contained only 2.2 percent moisture, 3.0 percent ash, and 0.6 percent sulfur. Yields of carbonization products in the 18-inch BM-AGA retort at 900° C. were: Coke, 70.9 percent; and (upon the basis of per ton of coal carbonized) gas, 10,900 cubic feet; tar, 13.5 gallons; light oil in gas, 2.80 gallons; and ammonium sulfate, 19.7 pounds. The coke was well-fused and less abradable than the average coke from high-volatile A coals tested previously. Blending with 20 percent Pocahontas No. 3 coal improved the physical properties of the coke, but no significant improvement was gained by increasing the proportion of this low-volatile coal to 30 percent. Oxidation tests indicated that the coking power of Powellton coal is less durable than that of Pittsburgh-bed coal. Powellton coal contracted 6.4 percent in the sole-heated oven at a charge density of 55.5 pounds per cubic foot; the blends containing 20 and 30 percent Pocahontas No. 3 coal expanded 3.2 and 5.1 percent, respectively, at the same charge density. The results indicate that blends of these coals should be used with caution in byproduct ovens.

Results of a study of the composition and carbonizing properties of Eagle-bed coal from a prospect shaft sunk from the Carbon Fuel Co. No. 7 mine working the Powellton bed at Carbon, Kanawha County, W. Va., were published.[66] The investigation included chemical and physical tests, carbonization tests by the BM-AGA method, expansion tests in the sole-heated oven, oxidation tests, and assays at low and high temperatures. Blends of Eagle

64/ Davis, J. D., Reynolds, D. A., Ode, W. H., and Holmes, C. R., Carbonizing Properties of Pocahontas No. 3- Bed Coal from Kimball, McDowell County, W. Va., and the Effect of Blending this Coal with Pittsburgh-Bed Coal: Bureau of Mines Tech. Paper 670, 1944, 35 pp.

65/ Reynolds, D. A., Davis, J. D., Ode, W. H., Brewer, R. E., and Holmes, C. R., Carbonizing Properties of Powellton-Bed Coal from Coal Mountain Mine, Guyan, Wyoming County, W. Va.: Bureau of Mines Tech. Paper 683, 1945, 44 pp.

66/ Reynolds, D. A., Davis, J. D., Ode, W. H., Brewer, R. E., and Birge, G. W., Carbonizing Properties of Eagle-Bed Coal from Prospect Shaft, Carbon, Kanawha County, W. Va.: Bureau of Mines Tech. Paper 691, 1946, 43 pp.

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and Pocahontas No. 3 coals were carbonized at 900° C.; one blend contained equal parts of Eagle and Powellton coal from the same mine. The Eagle-bed, high-volatile A bituminous coal fulfilled all chemical specification for gas and coking coals. Yields of carbonization products in the 18-inch retort at 900° C. were: Coke, 72.1 percent, and (upon the basis of per ton of coal carbonized) gas, 10,500 cubic feet; tar, 12.6 gallons; light oil, 3.44 gallons; and ammonium sulfate, 20.9 pounds. The yield of coke was 3.7 percent higher than the average yield from 32 high-volatile A coals carbonized previously, and the yields of gas and tar were lower than average for coals of that rank. Cokes obtained at all temperatures were well-fused. The 900° C. coke was fine-grained and moderately fissured and was more abradable than the average coke from high-volatile A coals. Blending with 20 and 30 percent of low-volatile Pocahontas No. 3 coal increased the yield and strength of coke and decreased the yields of gas, tar, and light oil. Blending with 50 percent of high-volatile A Powellton coal decreased the yield of coke, lowered the 1-1/2-inch shatter index, and raised the 1- and 1/4-inch tumbler indexes. Eagle coal contracted 21.2 percent in the sole-heated oven at a charge density of 55.5 pounds per cubic foot; the Powellton coal contracted 9.0 percent. A blend containing equal parts of these coals contracted 17.2 percent, and blends containing 20 and 30 percent of Pocahontas No. 3 coal contracted 11.8 and 5.9 percent, respectively. A blend of 35 percent Eagle, 35 percent Powellton, and 30 percent Pocahontas No. 3 coals contracted 3.6 percent. These results indicate that relatively large proportions of low-volatile coal could be blended with Eagle coal in commercial carbonization. Results of oxidation tests at 100° C. for periods of 4.8, 12.2, 20.0, and 33.2 days indicated that Eagle coal oxidized at a low rate and should have excellent storage properties.

Durability of Coking Power of Various Coals

The Bureau of Mines test for deterioration of coking power requires exposure to air in a steam-jacketed rotary drum at 100° C. of a large (400-pound) sample of stage-crushed 0- to 1/4-inch coal, followed by carbonization tests in 13-inch retorts at 800° C. of weighed portions (about 90 pounds each) of the coal after known periods of oxidation and determinations of the yields and quality of the resulting cokes and byproducts produced. The significant figure reported is the time of oxidation, in days, in air containing 20.93 percent of oxygen by volume, required to reduce the coke-strength index by 15 percent. This time is designated as T_{15} and is an expression of the "durability of coking power." The "durability of coking power" was determined for three low-volatile bituminous BM-AGA coals. These coals include Pocahontas No. 3 bed, Mead Nos. 2 and 3 mines, Pocahontas No. 4 bed, Mead No. 6 mine, and Beckley bed, Stanaford No. 1 mine, all from Raleigh County, W. Va. The "durability of coking power" value for Pocahontas No. 3 coal is 4.7, that for Pocahontas No. 4 coal is 7.3, and that for Beckley coal is 10.5 days. The value for Pittsburgh bed (Warden mine) high-volatile A bituminous coal from Allegheny County, Pa., is 28.5 days. It is evident from these results that the three low-volatile coals, in the order named, are considerably less resistant to exposure in storage than the high-volatile A coal.

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Oxidizing Properties of Bituminous Coals

Table 16 gives the sources and descriptions of the thirteen bituminous coals whose oxidizing properties were studied during the year. As noted in the table, a number of these coals were received washed, and therefore such samples did not contain their true natural bed moisture. In such instances, the classifications by rank were made from previously analyzed samples collected from the same mines. The washed 3/4- by 1/8-inch, Pittsburgh No. 8 bed, Piney Fork No. 1 mine coal was similar in composition to the washed 3/4- by 0-inch coal from the same source and was used to prepare five different closely sized fractions for study of the effect of size of coal upon the rate of oxidation. The second sample of Beckley-bed coal was prepared from the first, but dried in methane gas to a moisture content of less than 0.05 percent before oxidation.

Table 17 summarizes the relative tendencies of fifteen different samples of coal to heat spontaneously in storage. All of the samples except the sized fractions of Piney Fork No. 1 mine washed coal, designated by coal numbers 288.3, 288.2, 288.1, 288.5, and 288.4, were tested in stage-crushed 0- to 1/4-inch size. The screen analyses of these five fractions and of coal 263, which was prepared from the washed 3/4- by 0-inch Piney Fork No. 1 mine coal are given in table 18, which also shows the effect of size of coal upon the forms of sulfur present. The data in table 17 show that the tendency of bituminous coals to heat spontaneously in storage increases with decreasing rank of the coals. In general, the agreement by the two methods of test is reasonably good. The data for the Beckley-bed coal samples show that "dry" coal oxidizes less rapidly than the same coal containing the normal amount of moisture, which agrees with previous observations on many other coals that the unwashed or raw coal oxidizes less rapidly than the wet or washed coal from the same mine; for example, compare coals 262 and 263 in table 17. The increase in relative characteristic oxidation rates with decreasing size of coal is well-illustrated by the data for the five closely sized fractions of Piney Fork No. 1 mine coal. The screen analyses of these five fractions and of stage-crushed 0- to 1/4-inch coal 263 shown in table 18 indicate that coal 263 is intermediate in size range between coals 288.2 and 288.1. The data in parentheses in columns 4 and 5 of table 17 show that coal 263, taken as 1, oxidizes less rapidly than coal 288.2 and more rapidly than coal 288.1

Earlier studies by the Bureau of Mines have shown that the rates of oxidation of different-sized fractions of the same coal are proportional to the cube roots of their specific surface areas. Values for the cube roots of the specific surface areas were calculated for coals 288.3, 288.2, 288.1, 288.5, 288.4, and 263. Expressed in terms of unity for coal 263, the relative characteristic oxidation rates computed from specific surface areas were found to be of the same order of magnitude as the values given in parentheses in columns 4 and 5 of table 17. Lack of close agreement probably results from the several assumptions that must be made for certain data and constants used in calculating the specific surface area from the data of screen analyses.

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TABLE 16. - Source and description of coals tested for their oxidizing properties

State, county, town, and mine	Bed	Rank[1]	Agglomerating index[2]	Condition and size
ALABAMA				
Marion, Brilliant, Brilliant Nos. 2 and 7	Black Creek	(Hvab)	Cp	Washed, 1-1/2-inch slack.
Walker, Carbon Hill, New River	do.	(Hvab)	Cf	Washed, 3-inch by 1-1/2-inch.
Walker, Nauvao, Deepwater	do.	(Hvab)	Cf	Washed, 3-inch by 1-1/2-inch.
OHIO				
Jefferson, Piney Fork, Piney Fork No. 1	Pittsburgh No. 8	Hvab	Cf	Raw, 3/4-inch by 0-inch.
Do.	do.	(Hvab)	Cf	Washed, 3/4-inch by 0-inch.
Do.	do.	(Hvab)	Cf	Washed, 3/4-inch by 1/8-inch.
WASHINGTON				
King, Ravensdale, McKay	McKay	(Suba)	NAb	Washed, 1-inch by 3/32-inch.
Kittitas, Roslyn, N. W. I. Nos. 3, 5, and 9	Roslyn No. 5	(Hvab)	Cf	Washed, 1-5/8-inch by 0-inch.
Lewis, Centralia, Monarch No. 1	Foron	(Subc)	NAa	Washed, 1-3/4-inch by 5/8-inch.
WEST VIRGINIA				
Raleigh, Beckley, Stanaford No. 1	Beckley	Lvb	Cg	Raw.
Do.	do.	Lvb	Cg	Raw, dried before oxidation.
Raleigh, East Gulf, Mead Nos. 2 and 3	Pocahontas No. 3	Lvb	Cg	Raw.
Raleigh, East Gulf, Mead No. 6	Pocahontas No. 4	Lvb	Cg	Do.

1/ Rank according to A.S.T.M. Designation D 338-38: Lvb - low-volatile bituminous; Hvab - high-volatile A bituminous; Suba - subbituminous A; and Subc - subbituminous C. Ranks indicated by symbols in parentheses are based upon previously analyzed mine samples from the same mines.

2/ Cg - good caking; Cf - fair caking; Cp - poor caking; NAa - noncoherent residue; NAb - coke button shows no swelling or cell structure, and will pulverize under a 500-gram weight.

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TABLE 17. - Relative tendencies of coals to heat spontaneously in storage[1]

Coal No.	Bed and mine	Rank[2]	Relative characteristic oxidation rate at 212° F. when X=1[3]: Basis of dry, mineral-matter-free coal	Relative characteristic oxidation rate at 212° F. when X=1[3]: Basis of dry coal	Adiabatic calorimeter, ratio of self-heating rates on basis of dry coal at - 158° F.	Adiabatic calorimeter, ratio of self-heating rates on basis of dry coal at - 212° F.
281	Pocahontas No. 3, Mead Nos. 2 and 3	Lvb	0.57	0.55	-	-
280	Pocahontas No. 4, Mead No. 6	Lvb	.90	.87	-	-
90.1	Beckley bed, Stanaford No. 1 (dry coal)	Lvb	1.05	1.06	-	-
90	do.	Lvb	1.13	1.14	-	0.72
262	Pittsburgh No. 8, Piney Fork No. 1 (raw)	(Hvab)	1.19	.97	2.21	1.79
263	Pittsburgh No. 8, Piney Fork No. 1 (washed).	(Hvab)	2.13	2.00	4.09	2.58
288.3	do.	do.	1.35 (0.64)	1.29 (0.65)	-	-
288.2	do.	do.	1.89 (.89)	1.80 (.90)	-	-
288.1	do.	do.	3.14 (1.48)	2.98 (1.49)	-	-
288.5	do.	do.	4.64 (2.18)	4.42 (2.21)	-	-
288.4	do.	do.	9.36 (4.40)	8.84 (4.42)	-	-
260	Black Creek, Brilliant Nos. 2 and 7	do.	2.17	2.15	6.97	2.98
252	Roslyn No. 5, N.W.I. Nos. 3, 5, and 7	do.	2.80	2.43	3.17	1.53
255	McKay, McKay	(Suba)	12.14	11.57	15.79	8.71
251	Foron, Monarch No. 1	(Subc)	25.71	23.67	34.99	15.30

1/ The self-heating tendencies, except for values in parentheses in columns 4 and 5, are all expressed as ratios to washed Pittsburgh bed, Warden mine, coal (h28) = 1. Values in parentheses in columns 4 and 5 represent the ratios to Pittsburgh No. 8 bed, Piney Fork No. 1 mine, washed 3/4-inch by 0-inch (263). The absolute values for coal h28 are: column 4, 0.133 percent oxygen per day by weight of dry, mineral-matter-free coal after the dry, mineral-matter-free coal has consumed 1 percent of oxygen by weight; column 5, 0.127 percent oxygen per day by weight of dry coal after the dry, mineral-matter-free coal has consumed 1 percent of oxygen by weight; column 6, 0.783° F. per hour at 158° F.; and column 7, 3.29° F. per hour at 212° F. Corresponding absolute values for coal 263 are: column 4, 0.283; column 5, 0.254; column 6, 3.20° F. per hour at 158° F.; and column 7, 8.51° F. per hour at 212° F.

2/ Rank according to A.S.T.M. Designation D 338-38: Lvb - low-volatile bituminous; Hvab - high-volatile A bituminous; Suba - subbituminous A; and Subc - subbituminous C. Ranks indicated by symbols in parentheses are based upon previously analyzed mine samples from the same mines.

3/ Ratio of the characteristic oxidation rates of 0- to 1/4-inch coal at 212° F. in air after the dry, mineral-matter-free coal has consumed 1 percent of oxygen by weight.

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TABLE 18. - Screen analyses and sulfur forms of washed Pittsburgh No. 8-bed, Piney Fork No. 1-mine samples

Tyler standard screen scale sieves, opening, in inches[1]	Coal Numbers					
	288.3	288.2	263	288.1	288.5[2]	288.4[2]
Through 0.525 on 0.371	9.0					
Through .371 on .312 (2-1/2)	36.1					
Through .312 on .263 (3)	42.6					
Through .263 on .221 (3-1/2)	8.8					
Through .221 (3-1/2)	3.5					
On 0.185 (4)		34.3	34.2	8.9	4.2	0
Through 0.185 on 0.093 (8)		60.9	30.8	34.4	20.7	0
Through .093 on .046 (14)		1.7	15.0	37.5	29.2	.8
Through .046 on .0232 (28)			8.9			
Through .046 on .0164 (35)		.8		16.5	28.6	2.2
Through .0232 on .0116 (48)			6.0			
Through .0164 on .0069 (80)		1.2		1.8	9.9	5.9
Through .0116 on .0058 (100)			2.5			
Through .0069 on .0041 (150)		.5		.4	3.5	9.0
Through .0058 on .0029 (200)			1.2			
Through .0041 on .0029 (200)		.6		.2	1.5	9.0
Through .0029 (200)		0	1.4	.3	2.4	73.1
Totals	100.0	100.0	100.0	100.0	100.0	100.0
Sulfur forms (as-received basis), percent:[3]						
Sulfate	0.152	0.204	0.023	0.269	0.223	0.271
Pyritic	1.123	1.117	1.479	.983	1.024	.930
Organic	.946	.833	1.057	.955	.971	.978
Total	2.2	2.2	2.6	2.2	2.2	2.2

1/ Figures in parentheses designate Tyler mesh sizes.
2/ Coals 288.5 and 288.4 were prepared from reserve samples of coals 288.3 and 288.2, respectively.
3/ Analyses of coal 263 were made shortly after receiving the sample, whereas the washed 3/4- by 1/8-inch coal from which the five other samples were prepared had been in storage for 5 months. The storage before preparation of these five samples may account for their relatively higher proportions of sulfate and their lower proportions of pyritic and organic forms of sulfur, as compared with those of coal 263. The change in percentages of the different forms of sulfur with decreasing size of coal 288 is noteworthy.

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In connection with the cooperative study with the Southern Natural Gas Co. of the complete gasification properties of Alabama coals by the Lurgi process, using oxygen and steam, it was found that these coals were too strongly coking to be completely gasified before trouble resulted from coking of the coal charge while it was being fed into the bottom end of the vertical retort. A number of oxidation tests were made on two of the least strongly coking coals (from New River and Nauvao mines; table 16) in an effort to reduce the coking properties of these coals before charging them for gasification in the Lurgi retort. Oxidation was carried out in steam-jacketed, 50-pound-capacity, rotary-drum units using oxygen gas at 100° C. up to 100 and 141.62 hours. This oxidation treatment, while reducing the coking power, did not completely destroy the coking properties of these coals. The work is being continued for longer periods of time at higher temperatures.

Bureau of Mines Technical Paper 681, abstracted in last year's annual report,67/ was republished in condensed form.68/

Plasticity of Coal

Plastic properties of the 47 coal samples and blends described in table 19 were determined during the fiscal year. The samples were tested by either the Gieseler or Davis plastometer methods; two or more tests on the same coal were usually made by each method. A total of 187 tests was made, 65 by the Gieseler method and 122 by the Davis method.

The effect of oxidation was reflected in the plastometer tests made on a resample of high-volatile A bituminous Elkhorn No. 3 bed coal from Floyd County, Ky., which had been tested previously in March 1944. Gieseler maximum fluidity and Davis maximum resistance were reduced. Addition of 20 and 30 percent of low-volatile Pocahontas No. 3 bed coal from McDowell County, W. Va., to the Elkhorn coal reduced the maximum fluidity and increased the maximum resistance.

Beckley-bed coal from Raleigh County, W. Va., showed plastic properties characteristic of a low-ranking, low-volatile bituminous coal. Tests on 20:80 and 30:70 percent blends of this coal with high-volatile A Pittsburgh bed-coal indicated that the Beckley-bed coal would be a suitable low-volatile blending coal for making blast-furnace coke.

A blend of 49.5 percent of Eagle run-of-mine coal from the border of Fayette and Raleigh Counties, W. Va., and 50.5 percent of Pocahontas No. 3 slack from Raleigh County, W. Va., as prepared for coke-oven use by the Citizens Coke & Gas Utility, Indianapolis, Ind., was tested for its plastic properties. Excellent coking power of this blend was indicated by the 310 dial divisions per minute, maximum fluidity observed in the Gieseler test, and by the extremely high maximum resistance of 50.7 pound-inches in the Davis test.

67/ See footnote 27.

68/ Elder, J. L., Schmidt, L. D., Steiner, W. A., and Davis, J. D., Spontaneous Heating Tendencies of Coals: Am. Gas Assoc. Monthly, vol. 27, Sept. 1945, pp. 411-414.

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TABLE 19. - Description of coals and blends tested

Coal No.	Description
86	Elkhorn No. 3 bed, Wheelwright mine, Floyd County, Ky.
86A	80 percent coal 86 and 20 percent coal c75.
86B	70 percent coal 86 and 30 percent coal c75.
c75	Pocahontas No. 3 bed, Kimball, McDowell County, W. Va.
90	Beckley bed, Stanaford No. 1 mine, Raleigh County, W. Va.
90A	20 percent coal 90 and 80 percent coal 1-28.
90B	30 percent coal 90 and 70 percent coal 1-28.
1-28	Pittsburgh bed, Warden mine, Allegheny County, Pa. (washed)
257	49.5 percent Eagle run-of-mine coal from border of Fayette and Raleigh Counties, W. Va., and 50.5 percent Pocahontas No. 3 slack from Raleigh County, W. Va.
258	1.55 floats from Powellton bed, Powellton No. 2 mine, Fayette County, W. Va.
258A	80 percent coal 258 and 20 percent coal c75.
258B	70 percent coal 258 and 30 percent coal c75.
259	1.55 floats from Peerless and No. 2 Gas bed, No. 3 North mine, Fayette County, W. Va.
259A	80 percent coal 259 and 20 percent Pocahontas No. 3 bed coal b75.
259B	70 percent coal 259 and 30 percent Pocahontas No. 3 bed coal b75.
279	100 percent Peruvian coal, South America.
279A	20 percent coal 279 and 80 percent coal 276 (Lower Sunnyside bed, Sunnyside mine, Carbon County, Utah).
280	Pocahontas No. 4 bed, No. 6 mine, East Gulf, W. Va.
280-1-ox.	Coal 280, oxidized 3.8 days at 100° C.
280-2-ox.	Coal 280, oxidized 9.0 days at 100° C.
280-3-ox.	Coal 280, oxidized 14.2 days at 100° C.
281	Pocahontas No. 3 bed, Nos. 2 and 3 mines, East Gulf, W. Va.
281-1-ox.	Coal 281, oxidized 5.0 days at 100° C.
281-2-ox.	Coal 281, oxidized 11.0 days at 100° C.
281-3-ox.	Coal 281, oxidized 13.1 days at 100° C.
282	Pocahontas No. 3 bed, No. 2 mine, East Gulf, W. Va.
284	Core-drill hole No. 10-9, Coal Creek, Gunnison County, Colo., coal log 932 feet, 8-1/2 inches to 936 feet, 9-1/2 inches.
285	Core-drill hole No. 10-9, Coal Creek, Gunnison County, Colo., coal log 946 feet, 1 inch to 952 feet, 7 inches.
286	Core-drill hole No. 10-9, Coal Creek, Gunnison County, Colo., coal log 958 feet, 2 inches to 964 feet, 11 inches.
288	Pittsburgh No. 8 bed, Piney Fork No. 1 mine, Piney Fork, Jefferson County, Ohio.
-	Fleming Elkhorn gas coal, Elkhorn No. 4 mine, Kentucky.
	Alabama coals
	(All coals crushed to pass 1/2-inch, and screened fraction between 1/2-inch and 9 mesh used for tests.)
264	Carter bed, Snider mine, 1/2 mile S. E. of Cedar Cove, Tuscaloosa County.
265	Milldale bed, Abston mine, 8 miles E. of Peterson, Tuscaloosa County.
266	Brookwood bed, Abston mine, 8 miles E. of Peterson, Tuscaloosa County.
267	Mary Lee bed, Warrior River mine, 1-1/2 miles S. E. of Carbon Hill, Walker County.
268	Black Creek bed, New River mine, 1-1/2 miles S. E. of Carbon Hill, Walker County.
269	Mary Lee bed, Aldridge Shaft mine, 6 miles S. E. of Parrish, Walker County.
270	Black Creek bed, Deepwater mine, 1-1/2 miles S. E. of Nauvao, Walker County.
271	Thompson bed, Piper No. 2 mine, Piper, Bibb County.
272	Montevallo bed, Dogwood Nos. 1 and 3 mines, 1 mile N. W. of Underwood, Shelby County.
273	Mammoth Nos. 2 and 3 beds, Acmar mine (Colgate washer), 1 mile S. W. of Acmar, St. Clair County.
275	American bed, Virginia mine, 7 miles W. of Bessemer, Jefferson County.
	Special samples
DSC2	Grab sample from top bench of Pittsburgh bed, Shannopin mine, Bobtown, Greene County, Pa.
245A	85 percent lignite char (500° C.) from McAlester strip mine, 3 miles S. W. of Rockdale, Milan County, Tex., and 15 percent crude asphalt.
245B	85 percent Texas lignite char (400° C.) and 15 percent No. 4 asphalt binder.
245C	85 percent Texas lignite char (400° C.) and 15 percent No. 3 asphalt binder.
245D	85 percent Texas lignite char (300° C.) and 15 percent No. 4 asphalt binder.

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The 1.55 specific gravity floats from Powellton bed, Fayette County, W. Va., showed plastic properties typical of a coal of high-volatile A bituminous rank. Blending this coal with 20 and 30 percent of low-volatile bituminous Pocahontas No. 3-bed coal decreased the fluidity and increased the resistance. The 1.55 specific gravity floats from the Peerless and No. 2 Gas bed, Fayette County, W. Va., showed plastic properties similar to those obtained for the 1.55 floats from the Powellton bed; blending with 20 and 30 percent of low-volatile bituminous Pocahontas No. 3-bed coal produced the same characteristic changes as noted for the blend with Powellton floats.

The sample of 100-percent low-volatile bituminous coal from Peru, South America, showed plastic properties similar to those of other low-volatile coals tested in the BM-AGA Survey of American coals. Tests on a blend of 20 percent of this coal with 80 percent of high-volatile A Lower Sunnyside-bed coal gave results characteristic of such blends. The plastic properties indicate that the Peruvian coal would probably be suitable for blending purposes.

Three low-volatile bituminous coals, (1) Pocahontas No. 4 bed, No. 6 mine, (2) Pocahontas No. 3 bed, Nos. 2 and 3 mines, and (3) Pocahontas No. 3 bed, No. 2 mine, all showed similar plastic properties characteristic of coals of this rank. After several days' oxidation at 100° C. of the first two samples, their slight swelling tendencies disappeared, and after about 14 days' oxidation fusion was almost completely destroyed.

Three core-drill samples from Gunnison County, Colo., showed increasing fluidity and resistance in order of increasing depth of coal log, indicating that the coking properties should improve in that order. Judged from plasticity determinations, these samples offer promise of a new source of coking coal in Colorado. A series of core-drill samples, taken from another location in the same County last year, showed no fusion in either Gieseler or Davis plastometer tests at the normal rate of heating of 3° C. per minute.

Pittsburgh No. 8 bed coal from Jefferson County, Ohio, showed a low maximum fluidity and a high maximum resistance, indicating that it should make fair coke.

A sample of Fleming Elkhorn Gas coal from Kentucky was submitted by the Bureau of Mines, Synthetic Gas Production Laboratories at Morgantown, W. Va., for plasticity determinations. The data obtained indicate that this high-volatile A bituminous coal should make good coke.

In connection with a cooperative study with the Southern Natural Gas Co., 11 Alabama coals were tested for their plastic properties by the Davis-plastometer method to determine the suitability of these coals for complete gasification by the Lurgi process, which uses steam and oxygen at high and low pressures. Most of the coals proved to be strongly coking and would, therefore, offer too much resistance to the passage of gas through a heated coal column. The coals showing a long period of minimum resistance or maximum fluidity, a long plastic temperature range, and a low maximum resistance

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by plastometer tests appear preferable for gasification by the Lurgi process. Coals 268 and 270 appeared to have the most desirable properties and were studied extensively by this method of complete gasification.

Coal DSC2 from Greene County, Pa., is high-volatile A bituminous in rank and has a high sulfur content. This coal was used for experiments on the desulfurization of coal. Its plastic properties are typical of high-volatile A bituminous coals.

In a special series of four tests, 500°, 400°, and 300° C. chars were made from a Texas lignite and blended with various asphalt binders in the proportion of 85:15 of char to binder for plasticity determinations. Only slight fusion was shown by any of the blends. The 400° C. char with a heavy asphalt binder showed the most fusion, giving a resistance of 0.9 pound-inch in the Davis plastometer test.

A revolving drum for automatically graphing the resistance of a coal sample as it develops during the Davis plastometer test was devised and installed. After some adjustments were made in the gears and pulleys, the automatic recorder proved quite satisfactory and has been in continued use since early in the year.

Swelling Properties of Coal During the Coking Process

Bureau of Mines equipment for testing the swelling properties of coal during coking includes two vertical-slot ovens and a sole-heated oven, all electrically heated. The large vertical-slot oven uses a steel retort that holds 350 pounds of coal. The small vertical-slot oven requires no retort, as its heated surfaces are silicon carbide; the chamber holds 17 pounds of coal. These two vertical ovens are operated at constant volume, and the pressure exerted on the movable wall is measured. This pressure is the maximum to be expected on coke-oven walls. The sole oven is charged with 40 pounds of coal and is heated only at the floor. It is operated at a constant pressure of 2.2 pounds per square inch, and the percentage of expansion (or contraction) of the original thickness of coal charge is taken as the characteristic expansion index of the coal. Most duplicate tests in the sole-heated oven give expansion results that agree within 1 percent or less. This oven is useful for comparing different coals but does not show the maximum pressure to be expected on coke-oven walls.

Results from the small vertical oven are not reported at this time, as different test procedures and heating schedules are being used to determine the effect of these factors upon the maximum wall pressures and to develop a procedure that gives good agreement between duplicate tests. Thirty-nine tests were run in this small vertical oven during the year, and many data have been accumulated that may lead to a standard test procedure and correlation of results with either the large vertical oven or the sole-heated oven. One coal blend tested gave maximum wall pressures that correlated with the time required for the center of the coal charge to reach approximately 100° C. This time was less than 1 hour for the slowest heating rate

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or lowest wall temperature at charging. Other coals or blends may not show this same susceptibility to a slight change in heating rate or wall temperature at charging. For this particular coal blend, the effect of the heating rate through the preplastic range seems to have considerable effect upon the maximum wall pressures when the plastic layers meet. Table 20 gives the expanding properties of the coals tested during the year in the sole-heated oven and the large vertical oven.

A new sample of Pocahontas No. 3 coal (c75) for blending purposes was received. This coal was tested in the sole oven and expanded an average of 21.7 percent as compared with a sample from the same mine received and tested in 1942, which expanded an average of 24.4 percent.

It has been suggested that Beckley coal be used to augment the diminishing supply of low-volatile coals. As there are frequent occurrences of steep dips and rises in the Beckley bed, with thin coal on the crests of the rises and thick coal in the bottom of the dips, six special samples for expansion tests were taken from various parts of the mine in addition to a carbonization sample. Blends of 20 and 30 percent Beckley bed coal with 80 and 70 percent, respectively, of Pittsburgh-bed coal, did not show dangerous expansion pressures in the large vertical oven. However, the 30:70 blend was tested at a bulk density of 49.88 pounds per cubic foot and gave a maximum wall pressure of 4.4 pounds per square inch. High bulk densities using the 30:70 blend probably would give dangerous expansion pressures. Two sole-heated oven tests were made on Beckley coal with oxidized samples. After 18.4 days of oxidation at 100° C., the expansion was lowered to 3.6 percent. A dry sample oxidized for 8.2 days (and water equivalent to the normal moisture content of the coal then added) expanded 30.0 percent, or almost twice the expansion of the unoxidized sample of normal moisture content.

Coal blends were submitted by the Citizens Gas & Coke Utility, Indianapolis, Ind., to check the expansion of oil samples containing a high percentage of Pocahontas No. 3 coal. The first of these blends, coal 257, consisted of 49.5 percent Eagle coal from the border of Fayette and Raleigh Counties, W. Va., and 50.5 percent Pocahontas No. 3 bed coal from Raleigh County, W. Va. The second blend, coal 257A, consisted of 55.0 percent Eagle bed and 45 percent Pocahontas No. 3 bed coal. Blend 257A was received in the oiled condition; no unoiled samples were received. Both the oiled and unoiled blends were tested in the sole-heated oven and the large vertical oven. The large oven tests on the oiled samples all gave pressures greater than 4.4 pounds per square inch, or the limit of the gage. An attachment has since been added to the large vertical slot oven, so that pressures up to 5.0 pounds per square inch can be measured.

The 1.55 floats from two high-volatile A coals, Powellton and No. 2 Gas, were cleaned by a heavy-media process, and each sample was then blended with 20 and 30 percent Pocahontas No. 3 coal (c75). All of these blends were contracting in the sole-heated oven.

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TABLE 20. - Expansion-test results

Coal No.	Description	Sole-heated oven. Expansion at 55.5 lb. per cubic foot, percent	Large vertical-slot oven. Maximum wall pressure, lb. per square inch
c75	100 percent Pocahontas No. 3 bed	+21.7	
90	100 percent Beckley bed, carbonizing sample, third air course, first north, 150 feet inby No. 1 cross entry on right side	+15.9	
90A	20 percent Beckley bed and 80 percent Pittsburgh bed	- 3.6	1/0.8
90B 2/	30 percent Beckley bed and 70 percent Pittsburgh bed	+ 1.0	1/3.4
a90 2/	Mine sample, first right off first north mains 200 feet inby	+13.9	
b90 2/	Mine sample, fourth breakthrough between fourth right entries off eighth left	3/+12.6	
c90 2/	Mine sample, first breakthrough to right off No. 23 room off eighth left	4/+11.2	
d90 2/	Mine sample, first west mains 300 feet past property line in right entry right rib	+10.6	
e90 2/	Mine sample, No. 1 room first panel fifth north of eighth right 150 feet inby	+12.4	
f90 2/	Mine sample, No. 5 room off fifth left blind entry off No. 4 mains	+ 9.0	
906/	100 percent Beckley bed	+30.0	
907/	100 percent Beckley bed	+ 3.6	
257	49.5 percent Eagle bed and 50.5 percent Pocahontas No. 3 bed	+ 2.1	3.2
257	49.5 percent Eagle bed and 50.5 percent Pocahontas No. 3 bed (oiled sample)		5/4.4
257A	55 percent Eagle bed and 45 percent Pocahontas No. 3 bed (oiled sample)	- .7	1/, 5/4.4
258	100 percent Powellton bed (1.55 floats)	-17.2	
258A	80 percent Powellton bed and 20 percent Pocahontas No. 3 bed	- 5.3	
258B	70 percent Powellton bed and 30 percent Pocahontas No. 3 bed	- .5	
259	100 percent No. 2 Gas and Peerless bed (1.55 floats)	-14.7	
259A	80 percent No. 2 Gas and Peerless bed and 20 percent Pocahontas No. 3 bed	- 5.3	
259B	70 percent No. 2 Gas and Peerless bed and 30 percent Pocahontas No. 3 bed	- 1.9	
280 8/	100 percent Pocahontas No. 4 bed coal	+22.9	
280 8/	100 percent Pocahontas No. 4 bed	+22.7	
280 9/	100 percent Pocahontas No. 4 bed	+ 6.2	
280 10/	100 percent Pocahontas No. 4 bed	- 1.5	
281	100 percent Pocahontas No. 3 bed	+28.5	
281 11/	100 percent Pocahontas No. 3 bed	+20.4	
281 12/	100 percent Pocahontas No. 3 bed	+ 2.6	
281 13/	100 percent Pocahontas No. 3 bed	+ .9	
282 14/	100 percent Pocahontas No. 3 bed	+29.3	
282 15/	100 percent Pocahontas No. 3 bed	1/+29.8	

1/ Single test.
2/ Special expansion samples. 100 percent Beckley bed.
3/ Thick coal at bottom of slope.
4/ Thin coal at crest of slope.
5/ Not maximum wall pressure; unable to hold at constant volume conditions.
Time oxidized in air at 100° C., days: 6/, 8.2; 7/, 18.4; 8/, 3.8; 9/, 9.0; 10/, 14.2; 11/, 5.0; 12/, 11.0; 13/, 13.1.
14/ Pillar sample containing up to 1/2-inch lump.
15/ Pillar sample crushed to pass 1/4-inch screen.

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Although the chemical analyses, carbonizing properties, and plastic properties showed no significant differences for Pocahontas No. 3 and Pocahontas No. 4 bed coals, their expansion differed by 5.6 percent. Pocahontas No. 3 expanded more than the No. 4 coal, and its coke was slightly stronger. Prolonged oxidation of these two coals resulted in appreciable reduction in their expanding properties.

Blending Properties of Coals

For a number of years it has been the practice in the Bureau of Mines Carbonization Laboratory to make blending tests on all coals included in the Survey of Carbonizing Properties of American Coals. All low- and medium-volatile coals have been blended with Pittsburgh-bed coal from the Warden mine, which has been chosen as standard, and high-volatile coals have been blended with a low-volatile coal considered as standard. Several low-volatile coals have been used, but Pocahontas No. 3, from Kimball, McDowell County, W. Va., has been used in most cases. Blending proportions were chosen arbitrarily, being 20 and 30 percent of the low-volatile coal with 80 and 70 percent of the high-volatile. Results of these blending tests have been published with other data on the coals in question, but up to the present, no attempt has been made to summarize the results and generalize on the effect of blending upon quality of the coke and yields of byproducts. The data have been collected and summarized in Report of Investigations 3936.69/ The following conclusions were reached as a result of the summary:

The results of BM-AGA tests at 900° C. of 12 low-volatile, 8 medium-volatile, and 26 high-volatile coals and their blends were analyzed to determine the effects of blending upon the quality of coke and yields of byproducts. The low- and medium-volatile coals were blended with 70 and 80 percent Pittsburgh-bed coal; 5 low-volatile coals were blended with various high-volatile coals in the same proportions. The relative coking power of the individual coals and their blends was determined by comparing the shatter and tumbler indexes of the cokes. The following conclusions as to results in the BM-AGA retort were made from this study:

1. The average coking power of medium-volatile coals equals that of low-volatile coals. This equality holds true if they are carbonized separately or as blends with Pittsburgh-bed coal.

2. The most strongly coking medium-volatile coals coke fully as well as the most strongly coking coal of any rank when carbonized separately.

3. The coking power of medium- and low-volatile coals generally indicates their value as blending coals, although anomalous results are sometimes obtained where different high-volatile coals are used. The coking power of binary blends correlates with that of the higher rank coal more closely when medium-volatile coals are blended.

4. Pocahontas No. 4, Pocahontas No. 3, and Beckley are satisfactory low-volatile coals for blending with high-volatile eastern coals. Blends

69/ See footnote 62.

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containing Pocahontas No. 4 coal from No. 4 mine, Raleigh County, W. Va., yielded slightly stronger coke than similar blends containing Pocahontas No. 3 coal from No. 3 mine, Wyoming County, W. Va.

Hartshorne coal from the Quality mine, Sebastian County, Ark., is a very satisfactory low-volatile coal for blending with Henryetta coal from Oklahomas and Bevier coal from Kansas.

5. Blends of low-volatile coals yield more coke and less tar, light oil, and gas than blends containing equal proportions of medium-volatile coals. The yield of gas on the volume basis is approximately the same for blends of low-volatile and medium-volatile coals.

The above conclusions are made with respect to the coking conditions prevailing in the BM-AGA test retort. They are presented for the information of the coking industry in the hope that discussion and publication of results of plant operation will show how the results of the BM-AGA apparatus can be applied in the study of blending problems.

Bureau of Mines-American Gas Association (BM-AGA) Apparatus and Procedure

Apparatus and methods used in the Bureau of Mines-American Gas Association carbonization-test procedure were described in Bureau of Mines Monograph 5,70/ published after completion of the first 30 coals to be tested. Ninety coals have now been tested by the BM-AGA methods and gradually, over a period of 16 years, minor modifications in apparatus and procedure have been introduced as experience gained indicated. Radical changes have not been adopted, because any radical departure from procedures originally devised would interfere with comparisons of new test data with those already collected. Apparatus and procedure now used are described in Technical Paper 685,71/ issued during the year. The main improvements made in apparatus were (1) use of the electric furnace exclusively for heating the retorts (dispensing entirely with the gas furnace), (2) provision of water jackets for the light-oil scrubbers, in which the temperature is controlled automatically at 5° C., (3) better instrumentation, and (4) provision for studying the expanding and oxidizing properties of coals. Procedures have been modified only as to minor details in the interest of higher precision, and some tests have been added to give more complete information. For example, water displacement of the retorts is determined before and after carbonization tests. These tests show whether the coal expands strongly enough to deform the retorts and indicate roughly whether the coal is strongly or

70/ Fieldner, A. C., and Davis, J. D., Gas-, Coke-, and By-Product-Making Properties of American Coals and Their Determination: Bureau of Mines Mono. 5, 1934, 164 pp.

71/ Reynolds, D. A., and Holmes, C. R., Procedure and Apparatus for Determining the Carbonizing Properties of American Coals by the Bureau of Mines-American Gas Association Method: Bureau of Mines Tech. Paper 685, 1946, 35 pp.

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moderately expanding. Coals that do not expand strongly enough to deform the retorts should give no trouble in a byproduct oven unless charged at higher densities. More attention is now paid to the density of charge in the retorts, because charge density strongly influences expansion and physical properties of the coke to some extent.

Bulk-Density Research

The extent of packing coal particles (that is the bulk density of the coal) in coke-oven charges has a marked influence upon the behavior of the charge during coking. Fluctuations in the bulk density of coal cause varying charges per oven with resultant uneven plant operation and poor quality of coke output because of under- or over-coking of the charge. Excessively low bulk densities can cause fluxing of the brickwork of the oven walls when the ovens are being operated at the maximum flue temperature. Excessively high bulk densities can cause generation of excessively high pressures, which distort and damage the brickwork of oven walls.

A new test method,72/ called the dropped-coal method was developed by the Bureau of Mines for predicting the average density of coal in coke-oven charges, so that coke producers could have a reliable method for guidance in their effort to produce larger quantities of higher-quality coke in existing equipment. Figure 23 gives a comparison of actual bulk densities with those obtained by the new dropped-coal method, the standard A.S.T.M. method, and the cone method. The average density in the oven is the base or zero line in this illustration; the plotted points show how far samples of various oven charges deviated from the bulk density of the actual oven charge when the samples were tested by the various methods. In the range studied, the dropped-coal method developed by the Bureau followed the oven results with great fidelity, regardless of moisture content, oil treatment, or fineness of crushing of the coal, whereas previously existent methods were found to vary widely from the desired behavior. These studies confirmed the opinion of many operators that the older test methods did not give results that paralleled actual oven bulk densities closely enough to allow the use of correction factors. The complete report on this work gives detailed data on the comparative tests and also covers general aspects of bulk-density control as well as factors influencing the packing behavior of coal in full-scale ovens and test containers.

Gasification of Subbituminous Coal and Lignite

Research and development work on gasification of lignite and subbituminous coal in externally heated alloy retorts was continued on the small pilot plant at Golden, Colo., and on the commercial-size pilot plant at Grand Forks, N. Dak. The large plant was operated 1,400 hours during the year to produce 16,000,000 cubic feet of various grades of water gas from natural lignite.

72/ Auvil, H. S., Schmidt, L. D., and Graham, H. G., Control of Bulk Densities in Coke Ovens: Studies of Precision and Application of Various Testing Methods: Bureau of Mines Rept. of Investigations 3935, May 1946, 21 pp.

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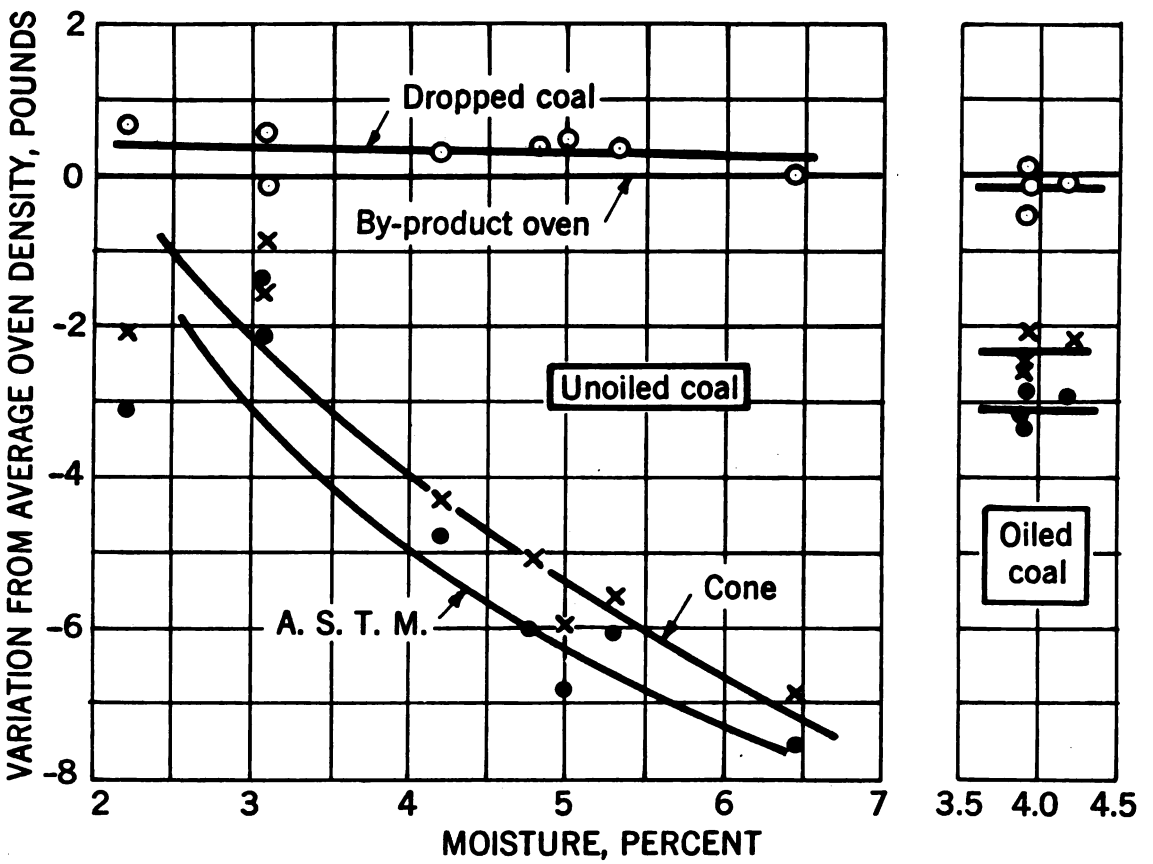

Figure 23. - Comparison between test results and the average density in byproduct ovens at various moisture levels. Oiled and unoiled coals.

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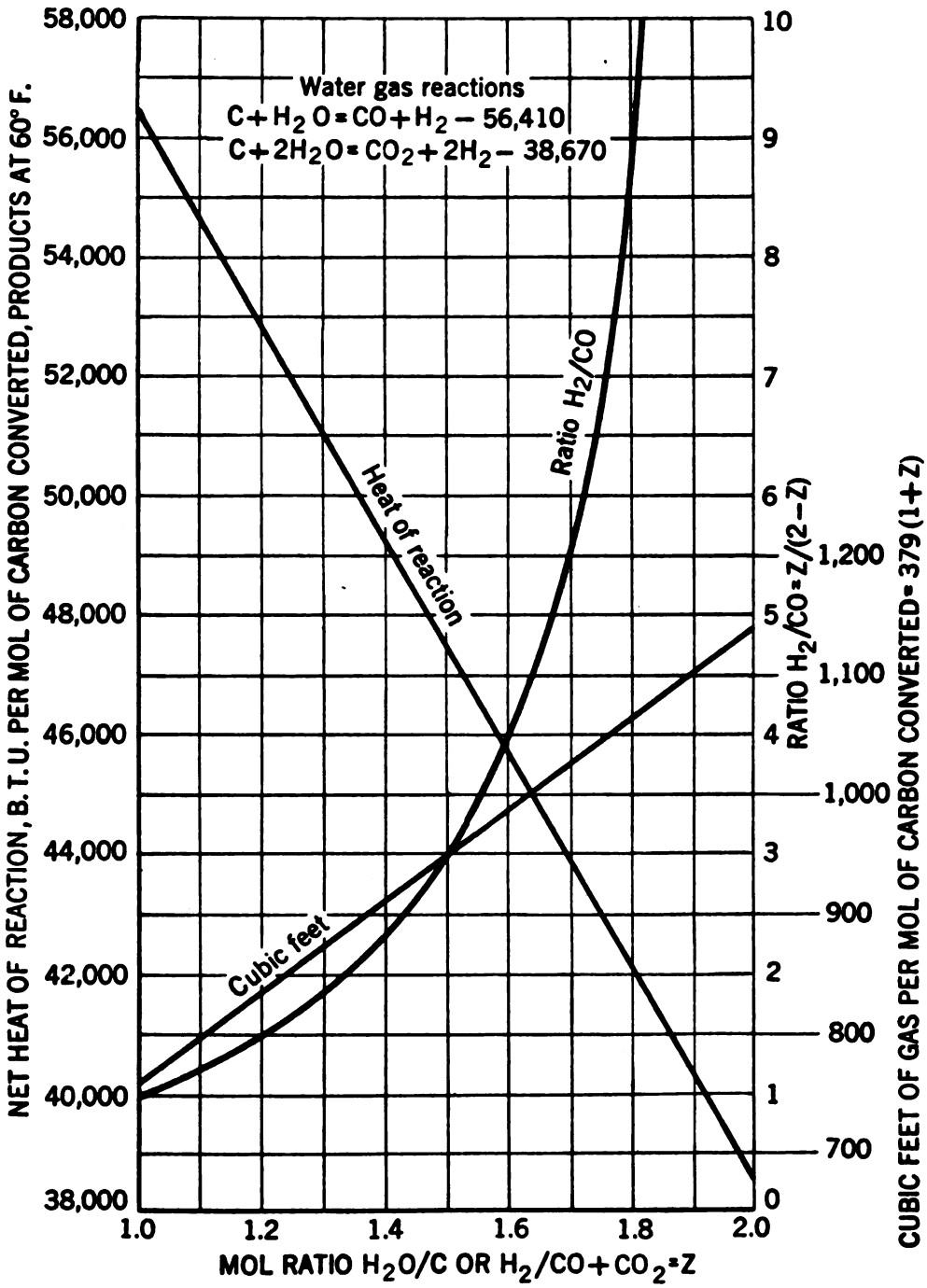

Figure 24. - Water-gas reactions.

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Research work was conducted at Golden to obtain data on the mechanism of the reactions encountered when coal is converted to gas in an externally heated retort and to observe the factors affecting capacity of the retort. It was demonstrated that the rate of gas formation can be increased from 66 to 81 cubic feet per hour per square foot of heated surface by decreasing the width of the reaction zone from 2.75 to 2.0 inches. Further studies were made on control of the composition of water gas by variations in temperature and in the concentration of steam, and the physical and chemical properties of high-ash char residues were determined.

The composition of water gases from low-rank fuels can be varied over a wide range. Figure 24 shows the change in gas composition and heat of reaction as the concentration of steam is increased. In the process under investigation, the $H_2/CO+CO_2$ ratio in the water gas, designated by "Z," can be varied from 1.35 to 1.80 by adjusting steam concentration and temperature. An important objective of pilot-plant tests has been to work out the operating problems involved in making these different grades of water gas. If the water gas is to be used for the manufacture of industrial hydrogen, the value of "Z" should be 1.7 to 1.8, and a high concentration of steam and relatively low temperatures are employed in making the gas. On the other hand, if the water gas is to be used for making synthetic gasoline, which needs a gas having a H_2/CO ratio of about 2.0, the apparent equilibrium or "Z" will be 1.3 to 1.5. Low steam concentration and relatively high temperatures are necessary for these conditions. The intermediate water gases that have an apparent equilibrium denoted by "Z" of 1.5 to 1.7 are the easiest type of gases to make in the externally heated retort, because the natural balance of hydrogen and carbon in the low-rank fuels favors this equilibrium at moderate temperatures. These intermediate gases probably will be useful as industrial heating gas. No tar is formed in this process, but the combined hydrogen appears in the final gas in the form of saturated gaseous hydrocarbons and illuminants.

The investigations during 1946 on the two pilot plants have solved many practical problems encountered in this new method of making water gas. The pilot plants were described in the 1945 report of research and technologic work on coal. It was found that both lignite and subbituminous coal will move freely through narrow reaction zones 2 inches wide. High rates of heat transfer through the metal wall in the range of 6,000 to 7,000 B.t.u. per hour per square foot were attained. It was further observed that the high-ash char residues are amenable to handling in simple mechanical and pneumatic devices. In the large plant, the unconverted char is removed continuously into a receiver and then extracted and transported from the machine by a gas jet, which conveys the char any desired distance from the generator. It was observed that operating problems increased as the concentration of steam was advanced.

The behavior of the alloy steel retort with respect to its life and cost is an important objective in operation of the plants. In the large plant, the vessel is 4 feet in diameter and 20 feet in length. It is suspended in a vertical furnace, which generates temperatures of 1,500° F. to

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2,050° F. by the combustion of a mixture of gas, air, and products of combustion. Thus far, during 1,400 hours of operation at 2,000° F. maximum temperature, the alloy retort shows no evidence of failure. It is believed that the retort will last several thousand hours, which will be ascertained during 1947, when the large plant will be operated 4,000 to 5,000 hours. Figure 25 shows the arrangement of the Grand Forks plant as it was operated during 1946.

This investigation has demonstrated the value of pilot-plant steps in the development of a process. The small plant incorporates the major features of a commercial-size plant on a reduced scale, and changes can be made quickly at low cost. The experience gained from its operation was useful in the design of the large plant. The size ratio of the two plants is 1:5. Tables 21 and 22 compare the performance of the plants on a unit basis. It is shown that the large plant is more efficient, whereas performance of the small plant indicates improvements that can be made on a larger scale. The operating characteristics of each plant are about the same, and experimental results obtained on coals tested in the small unit compare well with those obtained in the large unit.

A Report of Investigations describing the small pilot plant and other experimental work in connection with gasification of low-rank fuels has been published.73/

Gasification Processes Using Oxygen

Oxygen has been used extensively in Europe for the production of gas. The use of oxygen permits the continuous gasification of coal in gas producers yielding a substantially nitrogen-free gas superior in quality to normal producer gas. Oxygen gasification has not been utilized in the United States on a commercial scale because of the high cost of oxygen. As a result of recent developments in large-scale production of oxygen at lower cost and demands for gas for special purposes or from fuels not generally gasified, an intense interest in all gas-production processes using oxygen has been expressed by industry and research workers in the United States.

73/ Parry, V. F., Gernes, D. C., Goodman, J. B., Wagner, E. O., Koth, A. W., Patty, W. L., and Yeager, E. C., Gasification of Lignite and Subbituminous Coal Progress Report for 1944, (I) Carbonization and Gasification of Lignite in Laboratory Retorts; (II) Gasification of Lignite in Glover-West Retorts; (III) Gasification of Lignite Char Briquets in a Water-gas Machine; (IV) Gasification of Subbituminous Coal and Lignite in the Golden, Colo., Pilot Plant: Bureau of Mines Rept. of Investigations 3901, 1946, 59 pp.

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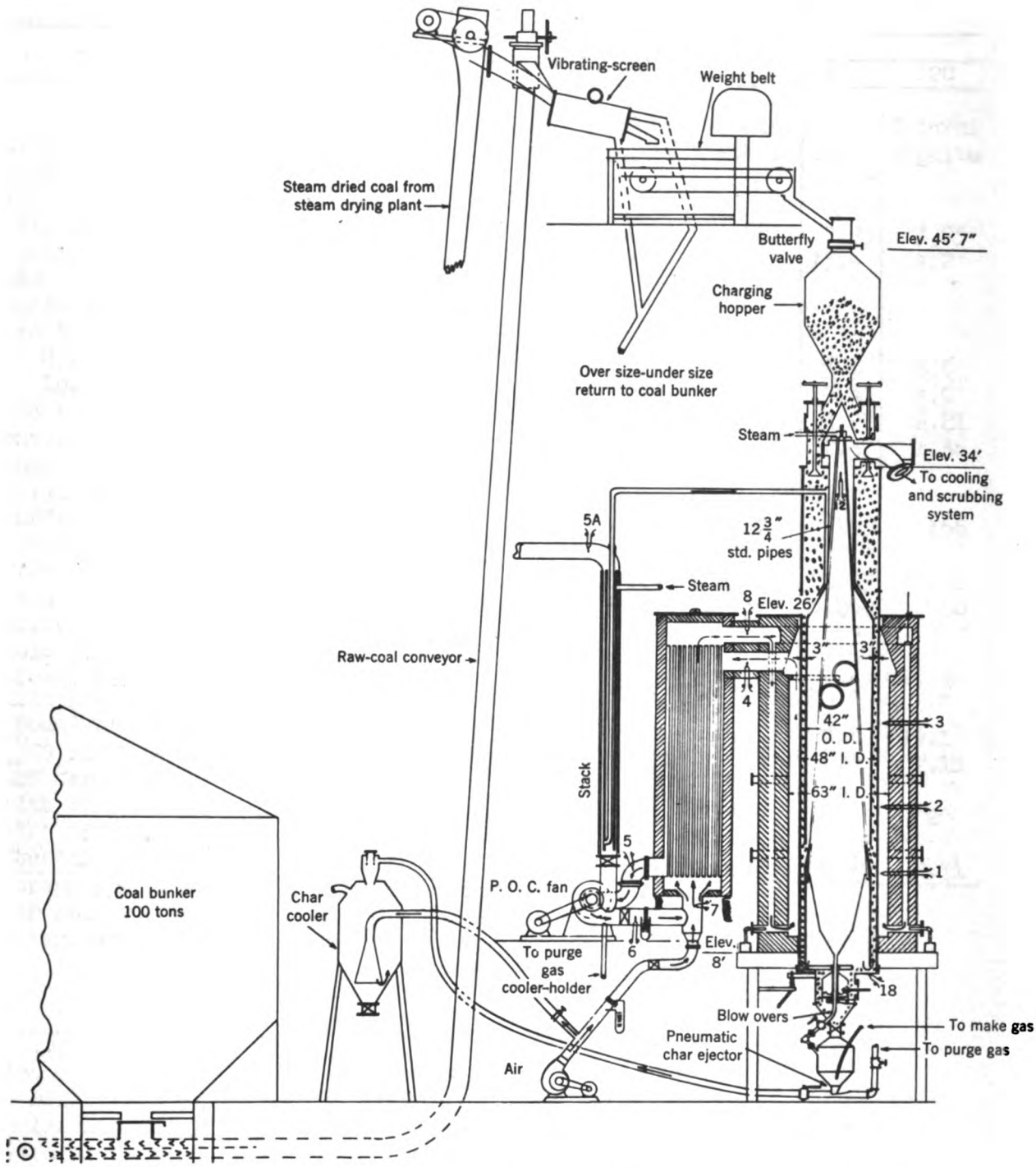

Figure 25. - U. S. Bureau of Mines pilot plant for gasification of lignite, Grand Forks, N. Dak., December 1945.

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TABLE 21. - Gasification of coal in small pilot plant, Golden, Colo.

	Test No.					
	5B	5C	8B	9D	10B	12C
Kind of coal	Subb.	Steam-dried lignite	Natural lignite	Wyoming subb.	Subb.	Natural lignite
Coal-charging rate, pounds per hour per square foot of retort surface	1.44	1.24	1.45	1.68	2.95	3,25
Percentage gasified[1]	76.6	94.4	89.8	74.7	73.4	64.2
Steam:						
Pounds per hour per square foot of retort surface:						
Upper annulus	4.0	3.8	1.9	2.6	3.1	3.6
Lower annulus	8.0	8.2	2.2	4.4	5.4	4.9
Used per mol of carbon gasified	7.0	6.25	2.93	3.65	2.02	2.81
Converted = Z[2]	1.75	1.65	1.49	1.47	1.37	1.36
Net heat used:						
B.t.u. per cubic foot of gas made	190	180	202	180	138	166
MB.t.u. per hour per square foot of retort surface	9.19	9.40	8.03	9.29	11.29	10.52
Average furnace temperature, °F.	1.540	1,605	1,690	1,730	1,800	1,750
Gas made:						
Cubic feet per hour per square foot, SGC[2]	46.6	50.5	38.7	50.3	81.5	63.3
Cubic feet of dry gas per pound of coal	32.9	41.4	27.0	30.4	27.1	19.1
H_2/CO ratio = R	7.18	4.73	2.72	3.04	2.0	2.11
Retort arrangement:						
Width of annulus, inches	3	3	3	3	2	2
Ratio of length of top to bottom annuli	2.45	2.45	2.45	2.45	2.79	2.11

1/ Percentage of carbon in coal converted to gas.
2/ Per mol of carbon gasified, $Z = H_2/CO+CO_2$.
3/ Saturated at 60° F. and 30 in. Hg.

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TABLE 22. - Gasification of coal in large pilot plant, Grand Forks, N. Dak.

	Test No.							
	Preliminary A	1B	2B	2E	3A	3B	3G	X[1]
Kind of coal	Lig.	Lig.	Lig.	Lig.	Lig.	Lig.	Lig.	Lig.
Coal-charging rate, pounds per hour per square foot of retort surface	3.00	3.46	2.43	3.64	2.00	3.30	2.69	3.71
Percentage gasified[2]	48.7	47.0	85.7	74.9	58.8	77.4	74.6	80.0
Steam:								
Pounds per hour per square foot of retort surface:								
Upper annulus	3.5	3.0	2.0	2.4	4.9	2.5	1.8	2.0
Lower annulus	2.9	9.7	3.2	4.0	4.6	4.6	2.3	4.0
Used per mol of carbon gasified	4.86	5.76	2.30	2.20	8.74	2.61	1.94	1.90
Converted = Z[3]	1.56	1.65	1.41	1.42	1.70	1.48	1.37	1.33
Net heat used:								
B.t.u. per cubic foot of gas made	144	130	126	127	201	123	121	110
MB.t.u. per hour per square foot of retort surface	6.47	6.54	7.80	9.84	7.66	9.23	7.22	9.35
Average furnace temperature, °F	1,825	1,670	1,770	1,790	1,345	1,785	1,810	1,825
Gas made:								
Cubic feet per hour per square foot, SGC[4]	46.1	50.3	61.7	77.6	38.2	74.9	59.9	85
Cubic feet of dry gas per pound of coal	15.1	14.3	25.0	20.9	18.8	22.3	21.8	22.5
H_2/CO ratio = R	2.37	2.58	2.44	2.48	5.43	2.71	2.18	2.0
Retort arrangement:								
Width of annulus, inches	4	4	3	3	3	3	3	2.5
Ratio of length of top to bottom annuli	2.02	4.03	2.26	2.26	2.11	2.11	2.11	2.0

1/ Desired conditions for 2,000-hour test.
2/ Percentage of carbon in coal converted to gas.
3/ Per mol of carbon gasified. $Z = H_2/(CO + CO_2)$ in the water gas formed.
4/ Saturated at 60° F. and 30 in. Hg.

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Results of the investigations in Europe by the members of the Technical Oil Mission were embodied in a report by the Bureau of Mines.[74] In this report, the oxygen-gasification processes were divided into four groups: (1) Processes in which fine fuel is gasified in a fluidized bed, (2) processes in which fine fuel is gasified in suspension, (3) processes in which fine fuel is gasified in a fixed bed, and (4) processes in which lump fuel is gasified in a fixed bed. The Winkler process was described as typical of the first group; the Koppers powdered-fuel process and the Schmalfeldt gasification process are typical of the second; the Lurgi pressure gasification process typifies the third; and the Thyssen-Galocsy and Leuna slagging generators are representative of the fourth.

As oxygen production is one of the prime considerations in connection with gasification processes, the most recent available results of oxygen production and its utilization in gas-making processes both at home and abroad were summarized in a more comprehensive publication,[75] which includes a brief history of the process, a discussion of the production of oxygen by the Linde-Frankl process, by electrolytic dissociation of water, and by the use of regenerative chemicals. The gasification processes described again included the Winkler, the Koppers powdered fuel, the Lurgi pressure gasification method of gasifying fine fuel in a fixed bed, and touched upon the Steere-Airco and Van Nuys methods of gasifying fine fuel in a fluidized bed or in suspension. In addition, methods of gasifying lump fuel in a stationary bed at normal pressures were described, and the results of operation of a pilot plant at Leaside, Ontario, were given. Slagging operations were covered in the descriptions of the Thyssen-Galocsy and the Leuna generators, which were observed in Germany.

LIQUEFACTION

Research on the production of liquid fuels from coal and lignite was begun by the Bureau of Mines in 1924. After collecting many fundamental data, the work was recessed until 1936, when a laboratory-scale experimental coal-hydrogenation plant capable of hydrogenating about 100 pounds of coal in 24 hours was designed and constructed. From 1936 to 1944, 16 coals were assayed, and the results demonstrated that low-ash and low-carbon coals were the most suitable varieties for liquefaction.

74/ Newman, L. L., Oxygen Gasification Processes in Germany: A.I.M.E. Tech. Paper 2116, 1946, 16 pp.

75/ Newman, L. L., Oxygen Production and Utilization in Gas-Making Processes: Report of Gas Production Committee, American Gas Association 1946 Proceedings, 24 pp.

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The passage of Public Law 290 in April 1944 permitted expansion of the Bureau's program in this field.76/ New buildings to house the research and development laboratories have almost been completed on a 12-acre portion of the Bureau of Mines Experimental Coal Mine and Explosives Testing Station property at Bruceton, Pa. While construction is going on at Bruceton, the Research and Development Division has been functioning in laboratory space provided at the Bureau's Central Experiment Station at Pittsburgh, Pa.

To carry the hydrogenation processes for the production of oil and gasoline toward commercial utilization, demonstration plants are to be built to furnish the necessary cost and engineering data for the development of a synthetic-liquid-fuels industry. New equipment and methods devised in the research and development program can thus be tested on a larger scale.

An extended study of some 206 proposed locations in 21 coal-producing States was made to select a plant site.77/ When it became apparent that some of the Government-owned synthetic-ammonia plants used in the production of munitions during the war would be closed down, it was decided that one of these would provide an ideal location. The War Department, acting through the Quartermaster-General, has made the synthetic-ammonia plant at Louisiana, Mo., available to the Bureau of Mines. The acquisition of this plant will result in a material saving in construction costs and will greatly speed the demonstration-plant program, as general facilities are immediately available, as well as much specialized equipment that can be incorporated into the demonstration plant.

Preliminary-flow diagrams and plot plants are now being prepared for this plant. Initial design is based upon about 200 barrels of oil per day, a figure that may be modified materially.

Reaction of Natural Gas and Steam

To evaluate proposals by industrial engineering companies to design and construct equipment for use by the Division at Bruceton, Pa., for synthesis-gas and hydrogen production, a study was made of the uncatalyzed natural gas-steam reaction. The proposed process was to heat the natural gas-steam mixture in a "pebble" furnace, in which alundum-clay spheres 1/2 inch in diameter are preheated by combustion of natural gas and then passed into the reaction vessel, where they transfer their heat to the

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76/ Fieldner, Arno C., Liquid Fuels from Sources Other than Petroleum. Statement in Investigation of Petroleum Resources (New Sources of Petroleum in the United States, Hearings before a Special Committee Investigation Petroleum Resources, United States Senate, 79th Cong., June 19, 20, 21, 22, and 25, 1945, pp. 336-349).
Fieldner, Arno C., Synthetic Liquid Fuels Program: National Petroleum News, vol. 37, No. 36, sec. 2, Sept. 5, 1945, pp. R-709-15.

77/ Unpublished report of the Secretary of the Interior to the 79th Cong. on the Synthetic Liquid Fuels Act from Jan. 1, 1945, to Dec. 31, 1945.

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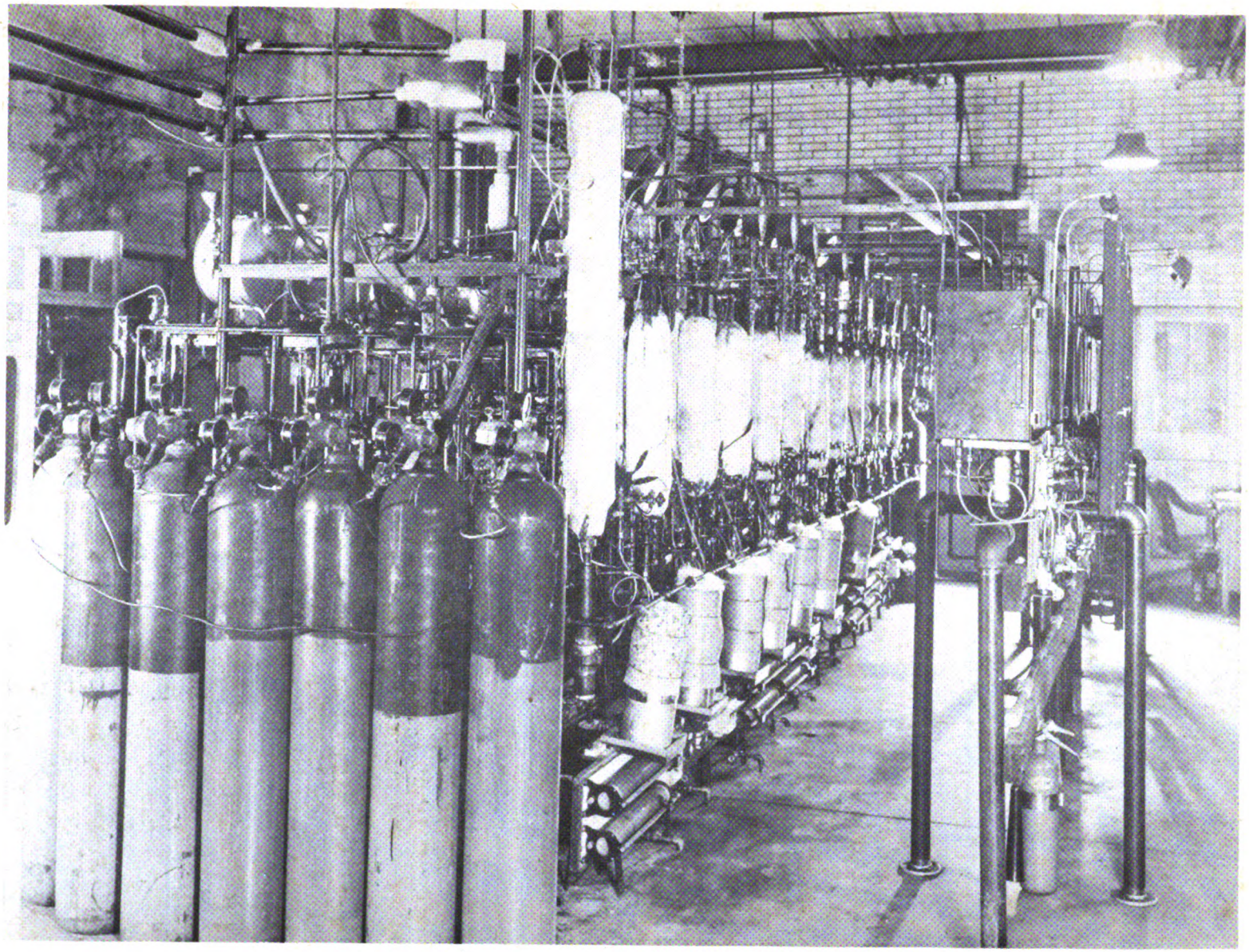

Figure 26. - Fischer-Tropsch catalyst testing units.

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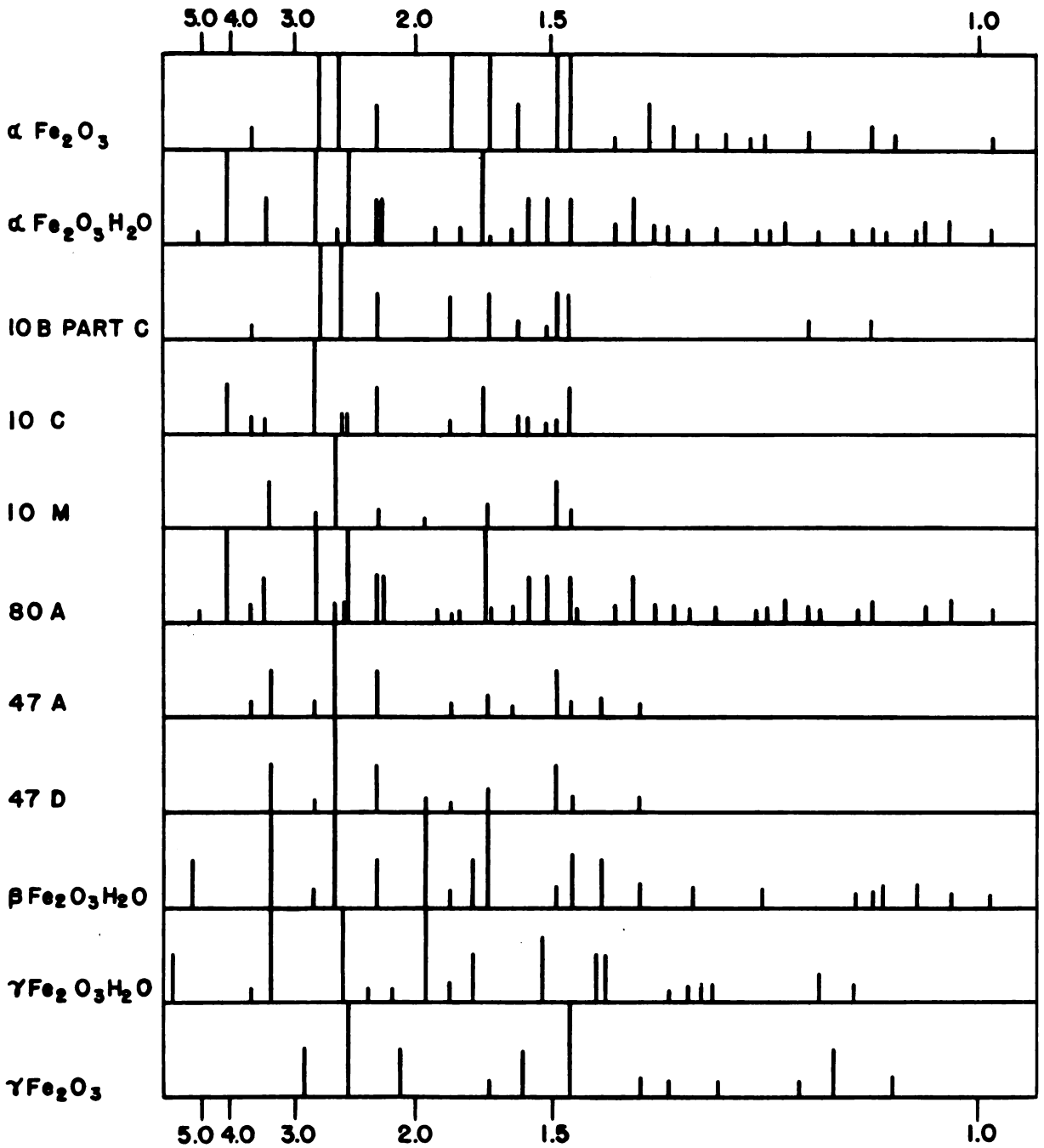

Figure 27. - X-ray diffraction powder patterns of various iron Fischer-Tropsch catalysts and the crystalline ferric oxides and ferric-oxide hydrates.

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natural gas-steam mixture. Because of uncertainties concerning reaction velocity and amount of carbon that might be deposited, the laboratory study was undertaken. Experiments were done in the temperature range 1,225° to 1,516° C. with steam-natural gas ratios of 1.5 to 5 and contact times in the range 0.21 to 4.6 seconds.78/ Even at the high temperatures, long contact times, and steam-natural gas ratio of 5, there is some carbonization of the natural gas. The extent of carbonization is dependent upon temperature, contact time, and partial pressure of steam.

Catalysts in Synthetic Liquid-Fuel Processes

A literature review was presented of the development of catalysts for the hydrogenation of carbon monoxide to yield hydrocarbons79/ and for the hydrogenolysis of coal and oil.

The chief aim of the Bureau of Mines catalyst testing program is to determine the influence on catalyst activity of conditions of catalyst preparation, pretreatment (such as reduction and carbiding), and mode of induction (starting the synthesis) for iron and cobalt catalysts. The tests were conducted in small converters containing about 45 cc. of catalyst in the shape of 1/8 by 1/8-inch pellets or granules. Figure 26 is a photograph of a battery of 12 units. At the right side of the photograph are the flowmeters that control and record the rate of flow of synthesis gas to the converters. The latter consist of 1/2-inch iron pipe filled to a depth of 12 inches with catalyst. The catalyst temperature was maintained by boiling Dowtherm in an electrically heated jacket surrounding the tube and controlling (automatically) the pressure on the Dowtherm.

The results of the catalyst-testing program are summarized as follows:

1. Active, durable, and easily regenerated iron catalysts can be prepared by maintaining special conditions of precipitation of iron hydroxide from ferric nitrate solutions and providing a small concentration (about 0.1 percent) of alkali, preferably potassium carbonate, in the finished catalyst. There is little if any advantage in two or three component iron catalysts such as iron-copper or iron-copper-manganese. This conclusion was arrived at also by the Kaiser-Wilhelm Institute für Kohlenforschung in Germany in publications cited later. (See Foreign Fuels, footnote 95.)

2. Excellent correlations have been observed between the X-ray diffraction patterns, magnetic susceptibilities of the iron catalysts, and their activity in the synthesis.80/ A comparison (see table 23 and figure 27) of the X-ray diffraction patterns of unreduced ferric oxide catalysts with

78/ Gordon, A. S., Uncatalyzed Reaction of Natural Gas and Steam: Ind. and Eng. Chem., vol. 38, 1946, pp. 718-20.

79/ Storch, H. H., Catalysis in Synthetic Liquid Fuel Processes: Ind. and Eng. Chem., vol. 37, No. 4, April 1945, pp. 340-351.

80/ Hofer, L. J. E., Peebles, W. C., and Dieter, W. E., X-ray Diffraction and Magnetic Studies of Unreduced Ferric Oxide Fischer-Tropsch Catalysts: Jour. Amer. Chem. Soc., vol. 68, No. 10, 1946, pp. 1953-1956.

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the diffraction patterns of the ferric oxide hydrates and of hematite (αFe_2O_3) showed that the inactive catalysts (with the exception of 47F) in the unreduced state, before use in the synthesis, all contained $\beta Fe_2O_3.H_2O$, and the active catalysts contained either $\alpha Fe_2O_3.H_2O$ or αFe_2O_3, or both, but no $\beta Fe_2O_3.H_2O$. Catalysts prepared from ferric chloride (the 47 series) or from ferric nitrate mixed with an appreciable quantity of potassium chloride (catalyst 10M) contained $\beta Fe_2O_3.H_2O$ and were inactive, whereas those prepared from pure ferric nitrate (the 10 series excepting 10M) were active. Catalyst 47F, which contains no $\beta Fe_2O_3.H_2O$ despite its preparation from ferric chloride, was inactive. It was shown that in this case $\beta Fe_2O_3.H_2O$ was initially present but that prolonged washing caused aging and conversion to αFe_2O_3. Comparisons of intensities (height of vertical lines) and wave lengths (Angstrom units on horizontal axis) given in figure 27 form the basis for the conclusions outlined above.

It should be noted that none of the iron oxides mentioned in the preceding paragraph is stable under the conditions of the Fischer-Tropsch synthesis on iron catalysts. The deactivating effect of precipitation in the presence of chloride ion is not due, therefore, to the presence of $\beta Fe_2O_3.H_2O$ itself, but to some characteristic which it imparts to the finished catalyst. Although chloride ion seems to have a small deleterious effect (see Foreign Fuels, footnote 95), the presence of $\beta Fe_2O_3.H_2O$ at any stage of the catalyst preparation greatly accentuates this deactivating effect. Active catalysts were obtained by precipitation from solutions containing cupric nitrate and ferric chloride; cupric chloride and ferrous chloride; and only ferrous chloride; but in each of these cases the precipitate at no stage contains any $\beta Fe_2O_3.H_2O$, as the presence of the bivalent ion insures a crystalline form other than $\beta Fe_2O_3.H_2O$.

The magnetic susceptibility of the catalysts was determined by measuring the force acting upon a sample suspended between the poles of an electromagnet with specially designed pole faces so as to obtain a uniform gradient between them. The magnetic balance used is shown in figure 28. It was calibrated at several field strengths with Mohr's salt (ferrous ammonium sulfate, hydrated crystals), the susceptibility of which at 27° C. was taken as 31.6×10^{-6}. The probable error of the measurements is in all cases $\pm 0.5 \times 10^{-6}$. All of the measurements were made at a field strength of 2,120 gausses. The magnetic susceptibility data of table 23 show that all the active 10-series catalysts, with the exception of 10M, and all the 80-series catalysts (prepared from ferric nitrate by precipitation with KOH solution instead of K_2CO_3 solution, as in the 10 and 47 series), had higher magnetic susceptibilities than the inactive catalysts.

3. Tests on the durability of iron catalysts showed that their useful life was only a few weeks at atmospheric pressure but at least several months at pressures of 5 to 7 atmospheres. The Kaiser-Wilhelm Institute experiments cited later (see Foreign Fuels, footnote 95) include tests that show that active iron catalysts have a useful life of more than 1 year at 15 atmospheres pressure.

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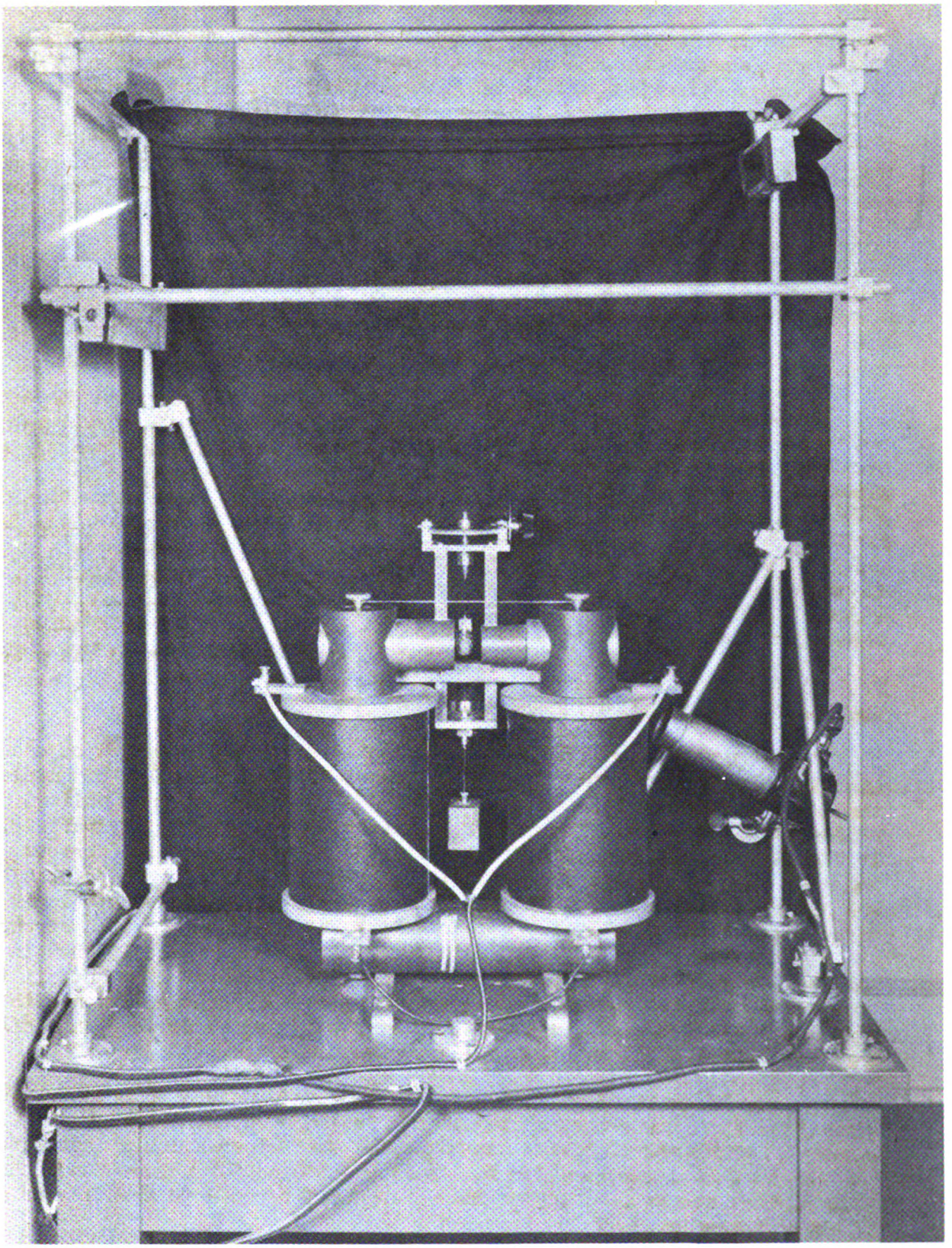

Figure 28. - Magnetic balance.

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TABLE 23. - X-ray diffraction, magnetic susceptibility, and activity data on unreduced iron catalysts

Catalyst	X-ray diffraction		Magnetic susceptibility x 10^{-6}	Activity			Operating pressure gage, p.s.i.
	Phases	Diffuseness[1]		Weeks tested	Optimum week	Yield[2]	
10A + 10B	αFe_2O_3 (probably)	X	150	15	2d	48.4	0
10B (pt. b)	αFe_2O_3	B	160	1	1st	16.7	0
10B (pt. c)	αFe_2O_3	B	150	2	1st	21.0	0
10C	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	135	5	3d	70.6	135
				1	1st	27.9	0
10D	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	210	4	1st	62.8	100
10E	αFe_2O_3	B	220	-	-	-	-
10F (pt. a)	αFe_2O_3	D	130	1	1st	34.5	0
10F (pt. c)	αFe_2O_3	D	180	3	1st	52.6	100
10G	αFe_2O_3	D	170	-	-	-	-
10H	αFe_2O_3	D	170	-	-	-	-
10I	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	140	-	-	-	-
10J	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	180	-	-	-	-
10K	αFe_2O_3	C	170	-	-	-	-
10L	αFe_2O_3	B	170	-	-	-	-
10M	$\beta Fe_2O_3.H_2O$	B	43	1	1st	<2	100
10O	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	130	-	-	-	-
47A	$\beta Fe_2O_3.H_2O$	B	44	1	1st	<2	0
47B	$\beta Fe_2O_3.H_2O$	C	56	1	1st	-	-
47C	$\beta Fe_2O_3.H_2O$	C	61	1	1st	<2	0
				1	1st	<2	100
47D	$\beta Fe_2O_3.H_2O$	C	61	1	1st	<2	0
47E	$\beta Fe_2O_3.H_2O$	C	40	1	1st	<2	0
47F	αFe_2O_3	A	130	1	1st	<2	0
47G	$\beta Fe_2O_3.H_2O$	C	56	-	-	-	-
80A	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	A	140	3	1st	63.9	100
80B	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	A	210	-	-	-	-

1/ A = sharp lines; B = diffuse; C = very diffuse; D = very, very diffuse; X = undecipherable.
2/ Grams of liquid hydrocarbons per cubic meter of synthesis gas.

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TABLE 23. - X-ray diffraction, magnetic susceptibility, and activity data on unreduced iron catalysts

Catalyst	X-ray diffraction: Phases	X-ray diffraction: Diffuseness[1]	Magnetic susceptibility x 10^{-6}	Activity: Weeks tested	Activity: Optimum week	Activity: Yield[2]	Operating pressure gage, p.s.i.
10A + 10B	αFe_2O_3 (probably)	X	150	15	2d	48.4	0
10B (pt. b)	αFe_2O_3	B	160	1	1st	16.7	0
10B (pt. c)	αFe_2O_3	B	150	2	1st	21.0	0
10C	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	135	5	3d	70.6	135
				1	1st	27.9	0
10D	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	210	4	1st	62.8	100
10E	αFe_2O_3	B	220	-	-	-	-
10F (pt. a)	αFe_2O_3	D	130	1	1st	34.5	0
10F (pt. c)	αFe_2O_3	D	180	3	1st	52.6	100
10G	αFe_2O_3	D	170	-	-	-	-
10H	αFe_2O_3	D	170	-	-	-	-
10I	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	140	-	-	-	-
10J	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	180	-	-	-	-
10K	αFe_2O_3	C	170	-	-	-	-
10L	αFe_2O_3	B	170	-	-	-	-
10M	$\beta Fe_2O_3.H_2O$	B	43	1	1st	<2	100
10O	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	B	130	-	-	-	-
47A	$\beta Fe_2O_3.H_2O$	B	44	1	1st	<2	0
47B	$\beta Fe_2O_3.H_2O$	C	56	1	1st	-	-
47C	$\beta Fe_2O_3.H_2O$	C	61	1	1st	<2	0
				1	1st	<2	100
47D	$\beta Fe_2O_3.H_2O$	C	61	1	1st	<2	0
47E	$\beta Fe_2O_3.H_2O$	C	40	1	1st	<2	0
47F	αFe_2O_3	A	130	1	1st	<2	0
47G	$\beta Fe_2O_3.H_2O$	C	56	-	-	-	-
80A	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	A	140	3	1st	63.9	100
80B	αFe_2O_3; $\alpha Fe_2O_3.H_2O$	A	210	-	-	-	-

1/ A = sharp lines; B = diffuse; C = very diffuse; D = very, very diffuse; X = undecipherable.
2/ Grams of liquid hydrocarbons per cubic meter of synthesis gas.

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4. X-ray diffraction patterns of cobalt-thoria-kieselguhr catalysts show that the unreduced catalyst is almost completely amorphous, the sharpest line in the pattern being that due to the quartz present in the kieselguhr. The X-ray pattern of the reduced catalyst shows the lines of cubic cobalt crystals. As the cubic form is unstable at room temperature, it is likely that the thoria and magnesia have an inhibiting effect upon the conversion of cubic to hexagonal cobalt.

5. A study was made of the porosity and surface areas of unreduced and reduced cobalt-thoria-magnesia-kieselguhr catalysts and of different types of kieselguhr.[81] Total surface area was measured[82] by determining the nitrogen absorption isotherms at -195° C. The number of molecules required to form a monolayer was calculated from the Brunauer, Emmett, and Teller Equation (Jour. Amer. Chem. Soc., vol. 60, 1938, p. 309), taking 16.2 Angstroms as the average cross-sectional area of the nitrogen molecule.[83] The nitrogen isotherms of unreduced cobalt catalysts are the usual S-shaped type, with only a slight amount of hysteresis, indicating that the pore structure consists of capillaries, most of which are larger than 500 Angstrom units in diameter. Most of the area is provided by the cobalt oxide-promoter complex and only little by the kieselguhr.

Kieselguhr, or diatomaceous earth, is a mineral composed of the skeletons of diatoms, which are microscopic plants. This residuum is essentially hydrous-amorphous silica. Table 24 presents data on surface areas and porosity of various kieselguhrs.

TABLE 24. - Properties of kieselguhrs

	Surface areas, m^2/gram	Pore volume, cc./gram		
		Micro	Macro	Total
German	14.9	3.22	3.62	6.85
Portugese	17.5	1.47	0.90	2.40
Filter Cel	22.2	2.81	3.32	6.13
Johns-Manville II	5.5	2.43	2.12	4.57
Hyflo Super Cel	1.9	2.32	1.77	4.09
Dicalite 911	29.3	2.22	1.41	3.63

Filter Cel, a marine diatomaceous earth from Lompoc, Calif., and Portugese kieselguhr (probably a fresh-water product) were found to be equally

81/ Anderson, R. B., Hall, W. K., Hewlett, H. D., and Seligman, B., Studies of the Fischer-Tropsch Synthesis. Part II. Properties of Unreduced Cobalt Catalysts: Jour. Amer. Chem. Soc. (In press.)
Anderson, R. B., Hall, W. K., Krieg, A., and Seligman, B., Studies of the Fischer-Tropsch Synthesis, Part III. Properties of Reduced Cobalt Catalysts: (In preparation.)

82/ Anderson, R. B., Improved Absorption Vessel: Ind. and Eng. Chem., Anal. ed., vol. 18, 1946, p. 156.

83/ Anderson, R. B., Modifications of the Brunauer, Emmett, and Teller Equation: Jour. Amer. Chem. Soc., vol. 68, 1946, pp. 686-90.

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effective as carriers for the cobalt-thoria-magnesia catalyst, and both were definitely superior to Hyflo Super Cel, which is prepared by alkali treatment and calcination of Filter Cel.

The surface areas of the catalysts are not additive with respect to the areas of the kieselguhrs used. Catalysts prepared from Hyflo Super Cel have lower areas than those prepared from natural kieselguhr, and in most instances the difference is greater than can be accounted for by assuming additivity of surface areas of the kieselguhr and the cobalt-promoter complex.

The bulk densities of the catalysts reflect the bulk density of the kieselguhr used in them. In granular catalysts, the bulk density varies directly with the bulk density of the kieselguhr, whereas the densities of the pelleted catalysts appear to be functions of the kieselguhr and of the pelleting, which is not always reproducible. It is interesting to note that the bulk volume occupied by an amount of granular catalyst containing 1 gram of kieselguhr is nearly equal to bulk volume of a gram of kieselguhr. When the catalyst is pelleted, the bulk volume is less than that of the kieselguhr that it contains.

In table 25 are given the surface areas and pore volumes of several unreduced cobalt catalysts in the granular and pelleted form. In every instance, the surface areas of pelleted catalysts are less than the corresponding granules. When the filter cake was slugged, that is, pressed into a very large pill, which was then ground and repelleted to produce harder and more reproducible pellets, the area dropped further. For example, 84.1 $m^2/g.$ for 108B granules, 76.0 $m^2/g.$ for small pellets, and 66.5 $m^2/g.$ for slugged pellets. These results are different from those found for the surface area of a rigid gel - such as silica-alumina used as an oil cracking catalyst - which was not changed by pelleting. The average pore diameter calculated for cylindrical pores from the pore volume divided by surface area are about 250 Å for pellets and 800 Å for granules. The MC15, which contains no kieselguhr, has pores of 330 Å diameter in a granular form. From the data presented, and others, the following picture of a cobalt catalyst is presented. The kieselguhr in a catalyst acts as a "brush pile" into which the cobalt complex may be placed without increasing the bulk volume. The cobalt complex is composed of clusters of much smaller particles than the kieselguhr, and these particles probably almost completely cover the kieselguhr, so that the total surface may be cobalt complex. Many of the pores are of the order of 200 to 1,000 Å in diameter.

As shown in table 25, the surface areas of unreduced cobalt catalysts are in the range 67 to 98 for the pellets and 84 to 110 square meters per gram for the granules. The activity of the catalysts listed in table 25 varies directly with the surface area of the unreduced catalyst. The presence of promoters in cobalt catalysts makes reduction more difficult and prevents sintering. The area of a cobalt-thoria-magnesia-kieselguhr catalyst, reduced at 400° C. in a rapid stream of hydrogen for 2 hours, was 37 square meters per gram. Approximately 40 percent of this surface was cobalt metal.

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TABLE 25. - Properties of unreduced cobalt Fischer-Tropsch catalysts

Catalyst	Form[1]	Kieselguhr	Area of kieselguhr, $m^2/g.$	Area of catalyst, $m^2/g.$	Percent cobalt	d_{Hg}[2]	d_{He}[2]	Pore Volume, cc./g.
Fischer (Cobalt-thoria-Kieselguhr):								
108B	G	Filter Cel	22.2	84.1	-	0.481	3.08	1.76
	P	Filter Cel	22.2	71.6	34.0	1.13	3.08	.56
	LP	Filter Cel	22.2	66.5	34.0	1.51	3.08	.34
Hall (Cobalt-Thoria-Magnesia-Kieselguhr):								
MC15	G	None		149.0		.676	3.80	1.22
89I	G	Hyflo Super Cel	1.9	83.6		.51	2.75	1.60
	P	Hyflo Super Cel	1.9	75.2	23.7	1.27	2.75	.43
89J	G	Filter Cel	22.2	104.1		.43	2.76	1.97
	P	Filter Cel	22.2	88.7	24.5	.97	2.77	.67
89K	G	Portugese	17.5	101.1		.61	2.75	1.28
	P	Portugese	17.5	98.2	24.6	1.20	2.75	.47
89U	G	German	14.9	109.3		.445	2.75	1.89
	P	German	14.9	86.2		1.10	2.75	.55
89V	G	D-911	29.3	101.9		.544	2.75	1.47
	P	D-911	29.3	77.6		1.31	2.75	.40

1/ G = granulated, P = pelleted, and LP = large pellets.
2/ d_{Hg} = specific gravity in mercury; d_{He} = specific gravity in helium.

Unreduced iron catalysts prepared by precipitation by alkali from aqueous solution have a higher surface area than unreduced cobalt catalysts and usually are of a gel-like structure. Upon reduction at 360° C., the areas of such iron catalysts decrease to about 5 percent of that of the unreduced catalyst.

6. Acid extraction and calcination of Filter Cel and alkali extraction and washing of the magnesia used in the preparation of the cobalt-thoria-magnesia-Filter Cel catalyst did not enhance its activity in the synthesis.

7. Several modes of induction (starting the synthesis after reduction of catalyst) of the cobalt-thoria-magnesia-kieselguhr catalyst were tested. No significant differences were observed owing to changes in starting temperature (usually 150° C.) or rate of increase of temperature. Induction at 100 pounds per square inch pressure of $2H_2+1CO$ resulted in lower activity during the first 400 hours of operation, as compared with similar induction at atmospheric pressure. Beyond 400 hours, the activity was the same for the inductions at the two pressures.

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8. Operation at atmospheric pressure of the synthesis on a Co-ThO_2-MgO-kieselguhr catalyst, for 1,500 hours, was followed by an additional 800 hours at 100 pounds per square inch pressure. The activity of the catalyst during the latter period was identical, within the limits of reproducibility of the tests, with that during the first 1,500 hours.

9. In the catalyst tests, a 15.8-mm. internal-diameter catalyst tube containing a 6.4 mm. outside diameter thermocouple well was used. The thickness of the annulus of catalyst was 4.7 mm. and its height was 30 cms. In this apparatus it was found possible to increase the space velocity (volumes of synthesis gas per volume of catalyst per hour) to about 500 without damaging the catalyst by sintering. The operating temperature increased from about 180° C. at 100 to 205° C. at 500 space velocity. The space-time yield of oil increased from 10 kilograms per cubic meter of catalyst per hour at 100 space velocity to 42 kilograms at 500 space velocity.

In general, pelleted catalysts produce more methane, C_1-C_4 and carbon dioxide than the granules (dry filter-cake, crushed and screened), as indicated in table 26 for runs 18 and 19 and 21 and 26. It will be noted that the temperature of operation at a given space velocity is considerably lower than for the pelleted catalyst, although when the temperatures are compared at a given flow per gram of catalyst the granules operate at slightly lower temperatures. It may be concluded that the variation of activity between pellets and granules is due to the decrease in surface area during pelleting. The change in the distribution of products is significant, but not too great. In most instances it would probably be desirable to pellet the catalyst to achieve the increased throughput, as the improved mechanical properties usually outweigh the undesirable effect of pelleting upon product distribution.

TABLE 26. - Granules vs. pellets data

Average temperature, ° C.	Contraction, percent	Space velocity 1/	Products in grams per cubic meter				
			CH_4	C_1-C_4	CO_2	H_2O	Liquids plus solids
	Run 21, pelleted 89J, 30.5 g. of catalyst						
186	72.0	112.0	20.4	33.0	7.9	190.6	101.4
	Run 26, granular 89J, 11.0 g. of catalyst						
194	63.0	97.4	15.4	28.0	5.2	158.5	84.6
	Run 19, pelleted 89K, 38.8 g. of catalyst						
176	72.7	101	21.6	37.0	12.7	180.7	90.8
	Run 18, granular 89K, 16.0 g. of catalyst						
188	73.5	100	17.6	30.2	8.0	177.4	89.5

1/ Volumes of synthesis-gas per volume of catalyst per hour.

10. Studies of the rates of carbiding of cobalt catalysts by carbon monoxide, of hydrogenation of the carbide, and of the synthesis have yielded

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some information concerning the temperature and pressure dependence of these rates. In experiments in which the catalyst testing converters described above were used, the temperature coefficient of the synthesis rate on a cobalt catalyst was measured by determining the space velocity necessary for 70 percent contraction at several temperatures in the range of 175°-200° C. From the temperature coefficients, the energy of activation was calculated to be 24 ± 2 kilocalories per gram molecular weight of $2H_2+1CO$ reacting.

Synthesis of Hydrocarbons from Hydrogen and Carbon Monoxide

Fischer-Tropsch Process Development

A brief review of the Fischer-Tropsch process, including some of the information contained in captured German documents, has been published.[84]

The synthesis of gasoline and Diesel oil from water-gas (a mixture of hydrogen and carbon monoxide) is accompanied by the evolution of a large amount of heat; namely, about 7,000 B.t.u. per pound of oil produced. The temperature of the reaction must be controlled within a narrow range of about 5° C. (9° F.) to avoid excessive methane production. These specifications necessitate rapid and efficient heat transfer from the catalyst surfaces to some cooling medium. The design of German converters involves the use of very narrow layers of catalyst and consequently very large amounts of steel (about 15 tons per barrel of oil per day). Recent process designs by United States oil-company engineers specify a turbulent bed of catalyst powder with heat-exchange tubes in the bed, or a heat exchanger outside the reaction zone, the catalyst suspension in the gas stream being passed from the converter through a heat exchanger and back into the converter. An even simpler process involving no internal metal heat-exchange surfaces is in process of development in the experimental plant of the Bureau of Mines. This process consists in injecting a cooling oil along with the feed gas. The boiling range of this cooling oil is so chosen that it will vaporize upon absorption of heat from the catalyst surface. Early experiments with a 12-inch catalyst bed 3 inches in diameter showed that very precise temperature control was obtained, but that the yield of oil per unit volume of catalyst per hour was erratic, and, although equal in amount to that of the German designs, the space-time yield was much lower than the American "fluid-flow" designs just described. Part of the difficulty causing the low and erratic yield has been traced to poorly reproducible catalyst-preparation procedure; but most of the trouble probably is caused by poor distribution of cooling oil and disturbance of the steady-state films of oil and gas on the catalyst surface by changes in cooling oil distribution.

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84/ Golumbic, N., Review of Fischer-Tropsch and Related Processes for Synthetic Liquid Fuel Production: Bureau of Mines Inf. Circ. 7366, 1946, 24 pp.

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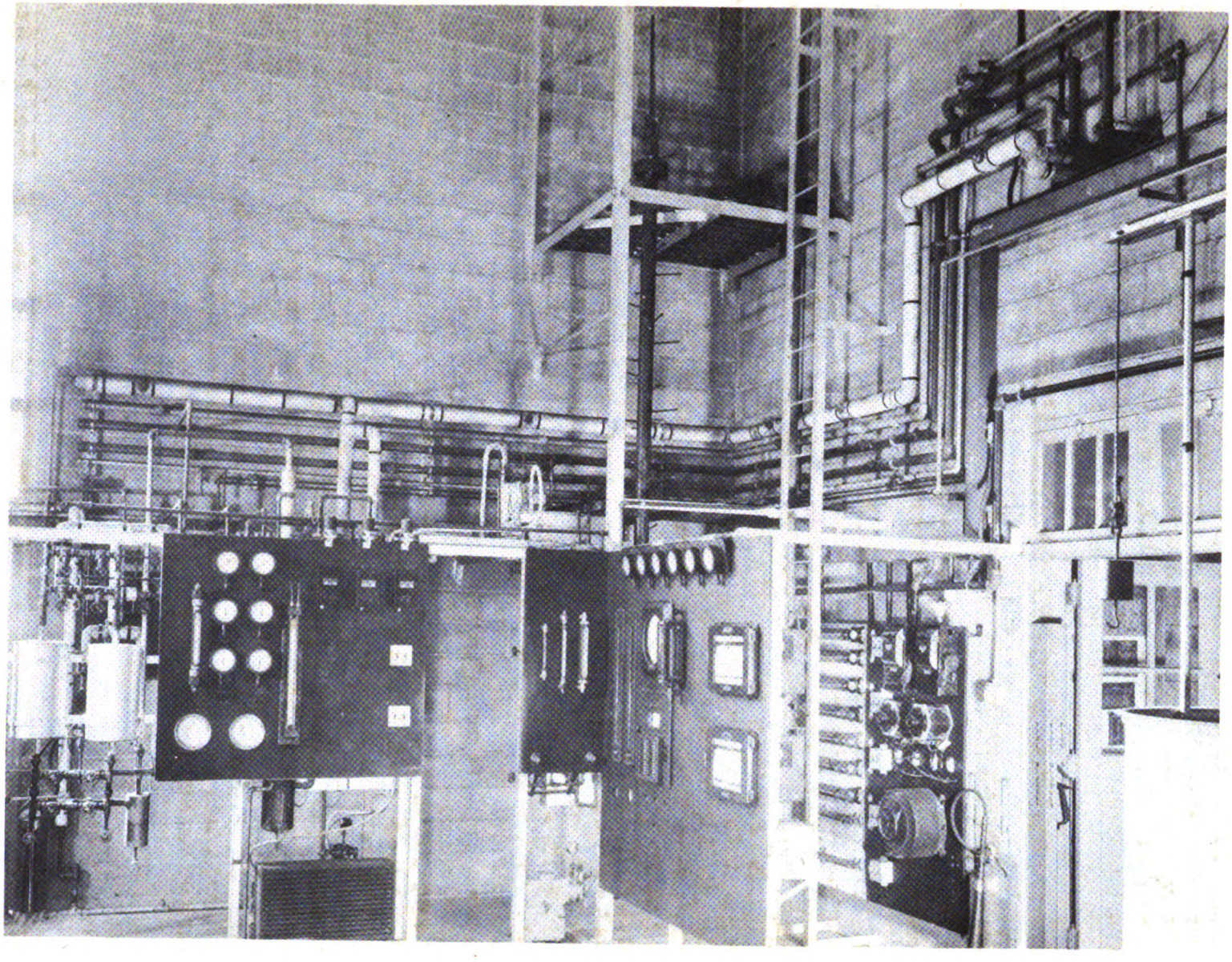

Figure 29. - Fischer-Tropsch experimental plant with internally cooled converter.

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The internally cooled converter previously described[85] was redesigned and rebuilt to accommodate a catalyst bed 3 inches in diameter by 8 feet deep and to provide adequate control of all operating conditions, such as pressure, temperature, rate of cooling-oil circulation, space velocity, and rate of recycling of end gas. Provision was made, also, for sample withdrawal at several points in the 8-foot column. A photograph of the partly completed unit is shown in figure 29.

Experiments with counter-current flow of cooling oil and a pelleted Co-ThO_2 Filter Cel catalyst showed that the maximum space velocity (volumes of synthesis gas per volume of catalyst per hour) permissible without flooding is about 200, whereas exploratory tests with co-current flow indicated that space velocities of 600 or larger could be used without flooding. An additional advantage of co-current operation was a much lower yield of methane and carbon dioxide than in the counter-current procedure. A recent co-current run operated for 200 hours at a space velocity of 100 at 183° C. and 100 pound per square inch pressure yielded about 11 kilograms of oil plus wax per cubic meter of catalyst per hour. The weight ratios of methane to oil plus wax and carbon dioxide to oil plus wax were, respectively, 0.20 and 0.16 for this run, as compared with 0.44 and 0.34 for a counter-current operation. Experiments at higher space-velocities in co-current operation are in progress.

The process-development laboratory has been enlarged during recent months as regards personnel, space, and equipment so as to conduct exploratory "bench" or laboratory-size experiments on several processes simultaneously. Three processes are being investigated; they are:

1. The liquid-phase catalyst-suspension process in which a finely divided catalyst is dispersed in a fraction of Fischer-Tropsch oil whose boiling point at operating pressure is maintained slightly higher than the desired operating temperature. Cooling may be accomplished by evaporation of oil or by circulation of a cooling fluid through an internal heat exchanger or by a combination of both types of cooling. Detailed plant have been completed for a 3-inch-diameter converter to have a maximum capacity of 2 liters of catalyst suspension and operate at 20 atmospheres pressure. Porous disks fabricated of sintered iron powder have been found suitable for distributing the feet gas. Special methods of producing extremely finely divided catalyst powders have been developed for use in this process.

2. Fluidized fixed-bed processes are being studied chiefly to develop such a process for production of a larger fraction of Diesel oil than is possible with the fluid-flow process now used industrially. To obtain some experience in control of important factors in fluidized catalyst operation, experiments in glass equipment were made with silica gel microspheres. One of the important variables is the manner in which the gas is introduced. Thus far, the best results have been obtained using a cone containing a

85/ Fieldner, A. C., Fisher, P. L., and Brewer, R. E., Annual Report of Research and Technologic Work on Coal, Fiscal Year 1944: Bureau of Mines Inf. Circ. 7322, 1945, 79 pp.

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fixed filter or gas distributing disk. Comparison of the quality of the fluidization is made on the basis of linear gas velocity, bed density, and bed resistance. Other variables to be studied are tube diameter, bed height, transverse baffles, and other dispersion devices, and particle size and its distribution.

3. The hot-gas recycle process, in which all of the heat of reaction is carried out of the reaction vessel by the sensible heat of the gas, involves very large recycle rates of the order of 50 to 100 parts of recycle gas to 1 part of fresh gas.

Coal Hydrogenation

Experimental Plant

The maintenance and mechanical design and assembly group of the experimental plant has been greatly improved as a result of greater availability of suitable mechanically adept personnel during the past year. Several additional automatic controls, such as high-pressure flowmeters, liquid-level controllers, and automatic alarm devices, have been installed. Reaction baskets were designed and used in most of the operations. These are fabricated of ordinary steel, and when coking occurs they are replaced by new baskets, thus avoiding the time-consuming and laborious decoking of converters. The mechanical group has been active, also, in design of special spray devices for atomizing coal-oil paste, of special paste and hydrogen preheaters, and in construction of models to study mechanical features of the coal hydrogenation process.

Further work was done on the production of a heavy ("Bunker C" type) fuel oil, using a pressure of 3,500 pounds per square inch. Under this pressure it was found possible to increase the throughput to about five times that at 1,000 pounds per square inch, using the same catalyst and temperature conditions in the converters. In the recent operations at the higher pressure, the hydrogen consumption was about the same as at 1,000 pounds per square inch. Data were obtained, also, for the increase in throughput with increasing reaction temperature. In the range of 420°-460° C., the throughput could be doubled for every 20° C. increase in reaction temperature. The effective contact times at different reaction temperatures and at 3,500 pounds per square inch pressure were as follows:

Temperature, ° C.	420	440	460
Contact times, hours	1.05	0.55	0.25
Hydrogen absorbed, percent of moisture and ash-free coal	4.6	5.3	5.1

Another objective of several runs in the experimental plant was to determine the importance of the extent of the gas to liquid interface in the first stage of coal hydrogenation. Two converters in series (see fig. 30) were packed with 1/2-inch coke, and the operation was conducted so as to produce chiefly fuel oil. The coke packing occupied about 50 percent of the converter volume. Coal-oil paste (see fig. 31 for photograph of paste-pump

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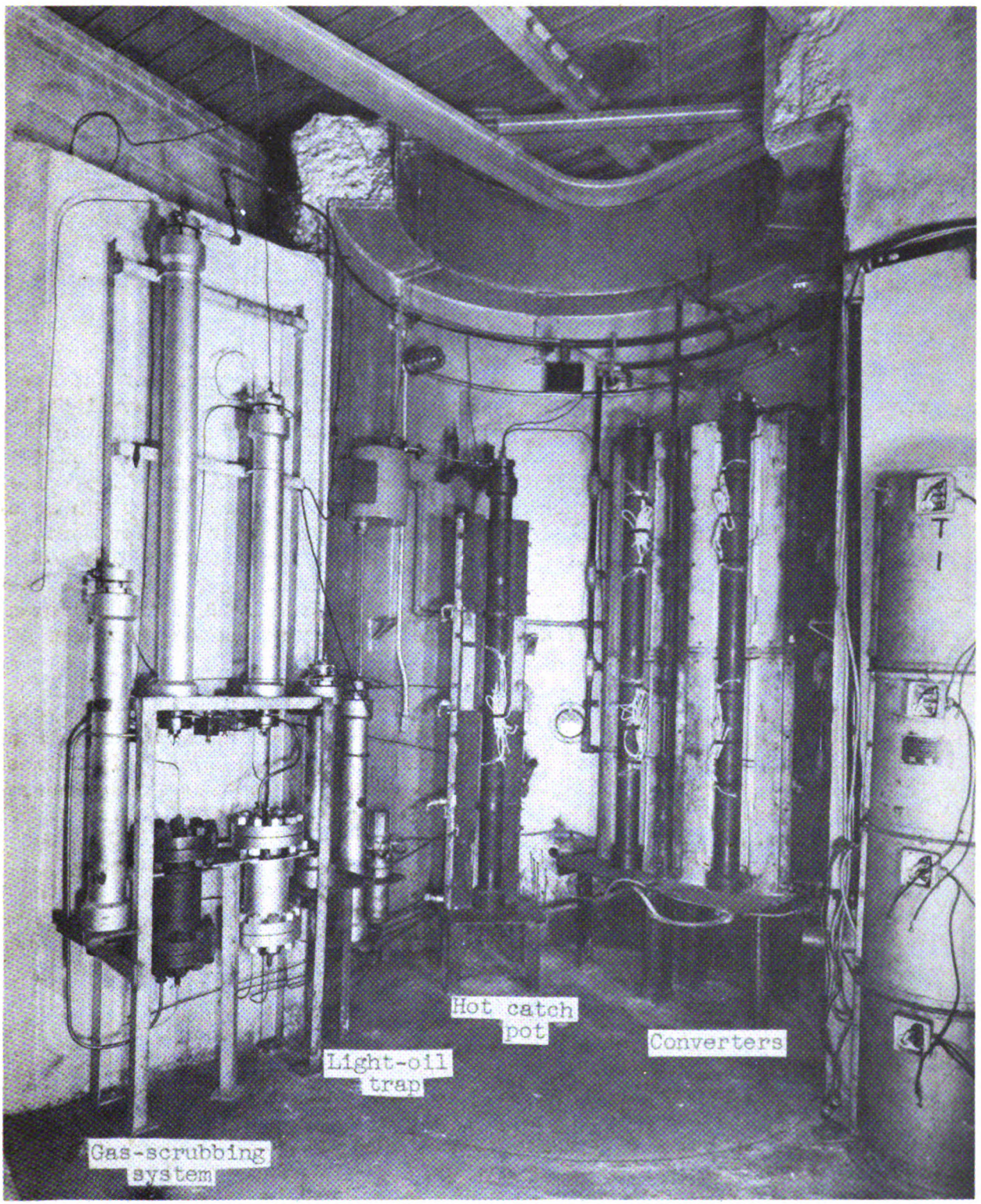

Figure 30. - Coal-hydrogenation experimental plant.

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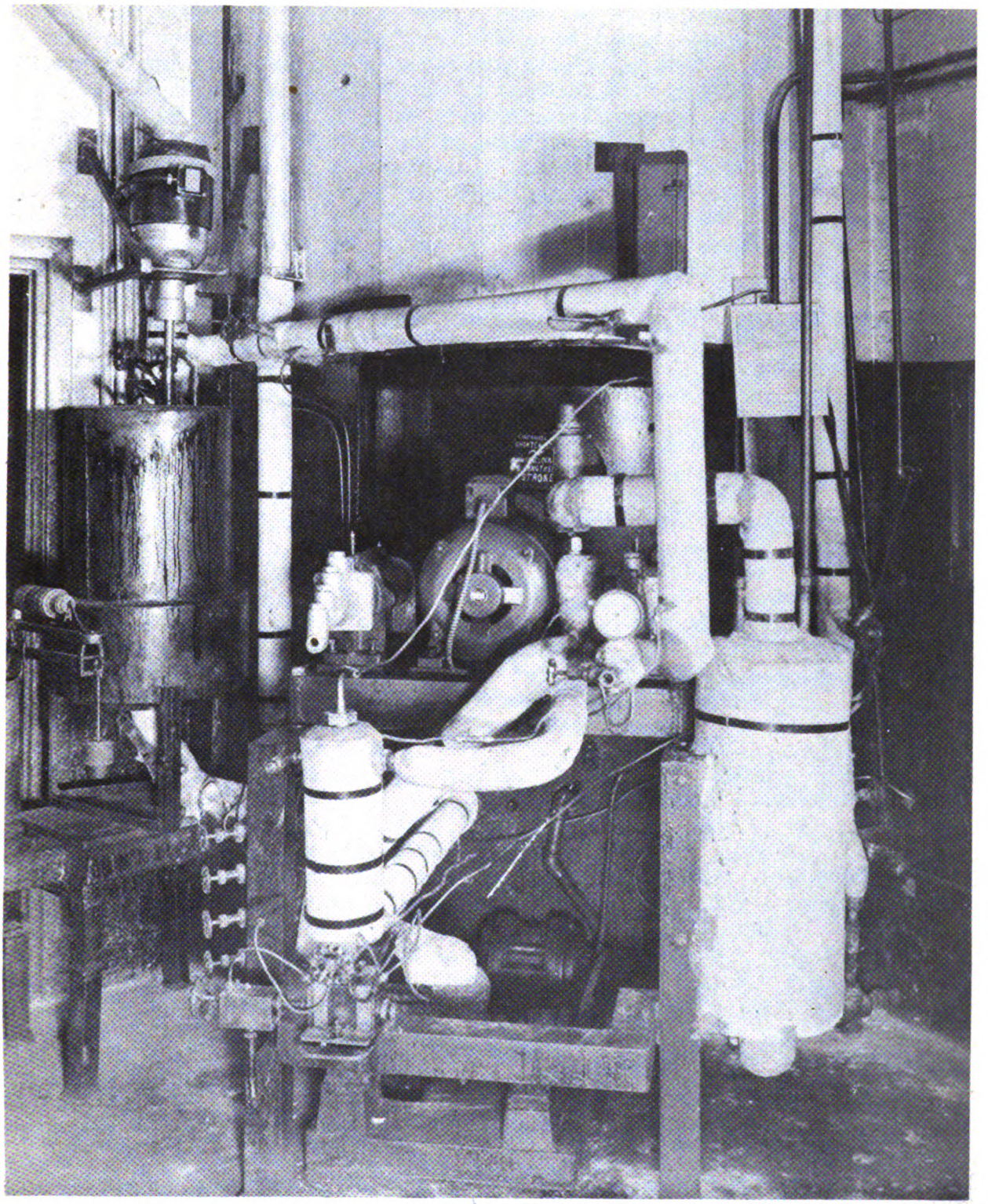

Figure 31. - Paste-pump assembly, coal-hydrogenation experimental plant.

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assembly) and compressed hydrogen were introduced at the bottom of the first converter and moved upward through the column of coke. The overflow from a standpipe in the first converter was conducted to the bottom of the second converter. It was found that only about one-half as much throughput could be maintained as was possible in an operation without the coke packing but otherwise identical with the operation just described. A smaller size (4 to 8 mesh) of coke packing did not result in any observable increase in throughput. It is apparent, therefore, that breaking up the size of the hydrogen bubbles by the coke packing did not increase the rate of coal hydrogenation. An appreciable decrease in the amount of hydrocarbon gas produced was noted in the packed converter system as compared with the unpacked.

To obtain a much larger increase in gas to liquid interface than in the system described in the previous paragraph, the preheated coal-oil paste was introduced at the top of column of coke, so that it would percolate down through the packing. In this fashion the entire surface of the coke, covered with a film of coal-oil solution, was exposed to hydrogen. The contact time at reaction temperature in this mode of operation was much smaller for a given throughput than in that described in the preceding paragraph. Even at one-half of the throughput used in the normal mode of operation, very serious operating difficulties were encountered with the percolation operation. After the seventh recycle batch, the amount of solids in the crude heavy oil was so high that centrifuging was difficult. Residual solids built up in the coke packing, so that the free space was reduced to such an extent that there was insufficient contact time for efficient hydrogenation. Addition of light oil to the pasting oil failed to improve the operation. Apparently the increased gas to liquid interface did not increase the rate of coal hydrogenation sufficiently to compensate for the greatly reduced contact time at reaction temperature in this operation.

The experimental plant was operated also in some preliminary runs on the possibility of using a fixed catalyst to accelerate the conversion of heavy oil to middle oil (or "gas oil"). It was thought that if the fixed catalyst were preceded by a bed of adsorptive material on which the nonvolatile oxygenated fractions of the heavy oil would be retained, rapid catalyst deterioration could be avoided and the nonvolatile constituents periodically dissolved by flushing the purification bed with a suitable solvent oil. It was found that a molybdic acid-on-alumina catalyst could be kept active for about 10 days in such a system, and that periodic flushings would be necessary every 4 or 5 days. Continuation of this research has been postponed until more information is available on the "steady-state" concentration of nonvolatile oxygenated compounds in liquid-phase coal hydrogenation and on their solubility in various light-oil fractions.

Experiments in Small Autoclaves

A series of experiments was concerned with the solvation and depolymerization of coal.[86] Tables 27 and 28 present data from various solvation

86/ Orchin, M., and Storch, H. H., Solvation and Hydrogenation of Coal: Ind. and Eng. Chem. (In preparation.)

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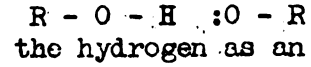
experiments. One atmosphere of hydrogen was present in all these experiments. In the experiments presented in table 27, the autoclave contents after reaction were transferred directly to centrifuge bottles, and the insoluble material was separated by centrifugation. The residue from the centrifugation was then extracted exhaustively with benzene, and the dried insoluble material was analyzed for ash to determine the percentage of liquefaction. In the experiments presented in table 28, the autoclave contents were poured, after reaction, into benzene, and the benzene mixture was centrifuged. The insoluble residue was exhaustively extracted and analyzed for ash to determine the percentage liquefaction. A comparison of experiments BK-98 and BK-99 in table 27 shows that under the same conditions o-cyclohexylphenol gives better liquefaction than tetralin. Comparison of BK-88 with G-68 indicates that tin sulfide and ammonium chloride have a deleterious effect upon the rate of liquefaction. Comparison of G-68 with BK-94 shows that the rate of liquefaction is unchanged with a small increase of the cyclohexylphenol-to-coal ratio. The results recorded in table 28 indicate that dilution of the product of the reaction with benzene causes the precipitation of additional benzene-insoluble material, resulting in lower liquefaction yields. Comparison of G-80 in table 28 with G-68 in table 27 indicates the magnitude of this difference. Comparison of G-74 of table 28 with BK-98 of table 27 indicates that the difference in working up the product affects the results with tetralin less than it does with o-cyclohexylphenol.

The results of these experiments can be correlated satisfactorily if one assumes that liquefaction in these experiments results principally from solvation of the coal by the solvent molecules. It is assumed that the coal structure is held together by secondary valence forces, which cause association of the coal polymer units. The association is assumed to be due to hydrogen bridging such as that that occurs in oxygen- and nitrogen-containing compounds. It is known that water, formic acid, acetic acid, and many other oxygen-containing compounds are associated. This association results from hydrogen bridging in which the



oxygen atom functions as an electron donor and the hydrogen as an electron acceptor to form a coordinate valence bond. Hydrogen bridging of this intramolecular type has important influence upon the physical properties of substances. Rodebush has stated in this connection that "it is remarkable that, as weak and relatively unstable a bond as the hydrogen bond is, it can affect physical strength and other important physical properties." It has been shown that natural polymers such as protein and cellulose depend for their structure to a considerable degree upon cross linkages of hydrogen bonds.

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TABLE 27. - Solvation experiments at 400° C.

Run No.	Vehicle	Catalysts	Time, hours	Ratio, vehicle / coal	Percent liquefaction, m.a.f.[1]
BK-87	Tetralin	2.50 percent SnS 0.55 percent NH_4Cl	1	4	58.4
BK-89	Tetralin	2.50 percent SnS 0.55 percent NH_4Cl	1	4	57.0
BK-98	Tetralin	2.50 percent SnS 0.55 percent NH_4Cl	1	4	54.7
BK-99	o-cyclohexylphenol	2.50 percent SnS 0.55 percent NH_4Cl	1	4	79.9
BK-88	o-cyclohexylphenol	2.50 percent SnS 0.55 percent NH_4Cl	1	4	74.5
BK-94	o-cyclohexylphenol	None	2	3	82.6
G-68	o-cyclohexylphenol	None	0.5	4	81.6
BK-100	o-cyclohexylphenol	None	0.5	4	82.7
BK-106	5-hydroxyl-1, 2, 3, 4-tetrahydronaphthalene	None	0.5	4	88.0

1/ In these experiments, the bomb contents were poured directly after the reaction into a centrifuge bottle without the addition of any benzene.

TABLE 28. - Solvation experiments at 400° C.

Run No.	Vehicle	Catalysts	Time, hours	Ratio, vehicle / coal	Percent liquefaction, m.a.f.[1]
BK-96	U.S.P. cresol	None	0.5	4	22.0
BK-97	U.S.P. cresol	None[2]	0.5	4	13.5
G-74	Tetralin	None	0.5	4	50.2
BK-93	o-cyclohexylphenol	None	2.0	1	30.5
G-80	o-cyclohexylphenol	None	0.5	4	63.7
G-76	50 percent tetralin, 50 percent o-cyclohexylphenol	None	0.5	4	56.0
G-70	p-cyclohexylphenol	None	0.5	4	60.2

1/ In these experiments, the bomb contents were washed out with benzene and the benzene suspension was centrifuged.

2/ 1,000 p.s.i. nitrogen was used in this experiment.

The striking effectiveness of o-cyclohexylphenol as a coal solvent may well be due to its ability to form hydrogen bridges. If the coal structure is held together by hydrogen bridges, then a substance which itself can form hydrogen bridges would be expected to solvate the coal. Tetralin lacks this property of forming hydrogen bridges, and so is less effective than o-cyclohexylphenol. If compounds capable of forming hydrogen bridges are, per se, good solvents for coal, one might expect the cresol would behave as a good solvent. Experiments BK-96 and BK-97 show, however, that cresol is a very

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ineffective solvent for coal. Its inferiority can, however, be satisfactorily explained on the basis that it has no hydrogen available to saturate the fragments of dissociated coal. Tetralin, which lacks a hydrogen-bonding group, is superior to cresol, probably because it can function as a hydrogen donor. That it, indeed, does this has been convincingly demonstrated by the fact that about 8 percent of the tetralin used in experiments G-74 was converted to naphthalene. Further substantiation of the necessity of hydrogen for good liquefaction is found in the experiments listed in table 29. In these experiments, high-pressure hydrogen was used. The results of experiment G-71 are particularly interesting. Though cresol is a very ineffective solvent without hydrogen (cf. BK-96, table 28), it produces excellent liquefaction in the presence of hydrogen. The hydroxyl group aids in the dissociation of the coal, and the high-pressure hydrogen saturates and stabilizes the dissociated fragments. The effect of catalysts in the hydrogenation experiments is in marked contrast to that in the solvation experiments. Experiments BK-92, GB-65, and GB-66 were run under identical conditions, except that increasing quantities of ammonium chloride were used. The increased ammonium chloride resulted in increased liquefaction and increased hydrogen consumption. The effect of tin sulfide is illustrated by comparison of G-67 with GB-66. In the former case, no tin sulfide was used, whereas in the latter the presence of 1 percent tin sulfide increased liquefaction about 12 percent.

TABLE 29. - Hydrogenation experiments at 400° C. and 1,000 p.s.i. initial hydrogen pressure

Run No.	Vehicle	Percent SnS	Percent NH_4Cl	Time, hours	Ratio, vehicle / coal	Percent liquefaction, m.a.f. basis
M-34	Tetralin	1.0	0.55	1	1	83.2
G-67	o-cyclohexylphenol	0.0	0.55	1	1	78.9
BK-92	o-cyclohexylphenol	1.0	0.0	1	1	80.0
GB-65	o-cyclohexylphenol	1.0	0.11	1	1	85.0
GB-66	o-cyclohexylphenol	1.0	0.55	1	1	90.7
G-69	p-cyclohexylphenol	1.0	0.55	1	1	88.4
G-75	Coal hydrogenation tar acids[1]	1.0	0.55	1	1	90.6
G-82	Pasting oil[2]	1.0	0.55	1	1	84.7
G-77	Petroleum ether, extract of pasting oil[3]	1.0	0.55	1	1	85.1
G-78	Same as G-77	1.0	0.55	1	1	84.4
G-71	U.S.P. cresol	1.0	0.55	1	1	91.7
BK-105	1, 2, 3, 4,-tetrahydro-5-hydroxynaphthalene	1.0	0.55	1	1	86.0

1/ The tar acid fraction from a light oil from the hydrogenation of coal was separated and the fraction b.p. 220-290°, used as vehicle.
2/ A typical pasting oil from a coal-hydrogenation plant.
3/ A petroleum ether extract of the above.

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From a consideration of the data, it is apparent that in the coal-hydrogenation process the coal structure is held together by associative forces of the hydrogen-bonding type, and that hydrogen-bonding solvents are effective vehicles for coal hydrogenation reactions. In the absence of high-pressure hydrogen, the solvent molecule must be capable of not only dissociating the coal, but it must also serve as a source of hydrogen to saturate the dissociated fragments of the coal structure.

Some of the evidence that suggests that the coal-liquefaction process involves the breaking of hydrogen bonds in the coal is listed below.

1. The magnitude of the difference in liquefaction results when the two different methods of working up the bomb contents are used in the cases of tetralin and o-cyclohexylphenol as vehicles. This results from the greater quantity of material precipitated upon dilution with benzene of the coal solution in o-cyclohexylphenol, as compared to the precipitate obtained when the coal in tetralin solution is diluted with benzene.

2. The precipitate obtained by dilution with benzene of the coal in o-cyclohexylphenol has a higher oxygen content than the corresponding precipitate obtained from tetralin.

3. Catalysts do not improve the liquefaction when solvents are used without high-pressure hydrogen.

4. Increasing the vehicle-to-coal ratio increases the liquefaction in solvation experiments, whereas under certain conditions the increase has no effect in hydrogenation experiments.

5. U. S. P. cresol per se is a poor solvent for coal, but produces excellent liquefaction in the presence of high-pressure hydrogen.

6. The concomitance of liquefaction and oxygen elimination suggests oxygen is involved in the linking of the coal polymer.

7. Catechol has been shown to produce low molecular weight fragments from humic acid by the simple process of solution.

8. Solutions of coal in o-cyclohexylphenol are very stable and can be kept without apparent change.

Identification of Products of Coal Hydrogenation and Study of Typical Reactions Involved in the Process

Organization of the personnel and erection and standardization of equipment for the precision distillation laboratory were completed. The equipment includes a 3-inch by 20-foot, stainless-steel, Foster-Wheeler distillation column with Stedman packing and a 25-gallon boiler. This is to be used for the initial distillation of synthetic liquid-fuel oils. The fractions obtained by the use of this still will be subjected to "type-separations"

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by selective adsorption on silica gel. The pure types of hydrocarbons are then distilled in one of several vacuum-jacketed precision glass columns. For the identification of the pure fractions obtained by use of these columns, their characteristic physical constants (freezing point, refractive index, etc.) and the melting points of pure, crystalline derivatives are measured. Most aromatic polynuclear compounds such as are contained in coal hydrogenation oils form complexes with picric acid and with 1, 3, 5,-trinitrobenzene. These complexes usually are crystalline solids with sharp melting points. The use of picric acid and trinitrobenzene for the caracterization of high molecular weight polynuclear compounds suffers from the disadvantage that the hydrocarbon is relatively only slightly soluble and often precipitates before the complex, or it is precipitated in excess on the complex compound. It was found that 2, 4, 7,-trinitrofluorenone is a much better reagent, and the molecular complexes of this compound with 26 pure polynuclear hydrocarbons were prepared and their melting points determined.87/

For the characterization of coal-hydrogenation oils boiling above 300° C., precision vacuum distillation is desirable. Because no satisfactory standards have been established for distillation efficiencies of vacuum stills, a search is being made for a two-component mixture of oils which is suitable for such standardization work. Vapor pressure data were obtained for cyclohexylcyclopentane, n-dodecane, and different mixtures of these two compounds. Similar data are being obtained for tridecane and dicyclohexyl.88/

The presence in coal hydrogenation and coal carbonization liquid products of polynuclear hydrocarbons with several fused rings sometimes has been used as evidence that such structures exist in the original coal. The possibility that polynuclear hydrocarbons containing many rings may be formed by dehydrogenation and cyclization of polynuclear hydrocarbons containing a smaller number of rings, or of the alkyl derivatives of such smaller ring systems, has been investigated, and several such syntheses have been demonstrated.89/

Characterization of Light Oil

Preliminary characterization of the light oil obtained as a minor product in the low-pressure hydrogenation of Bruceton coal was accomplished by

87/ Orchin, M., and Woolfolk, E. O., Molecular Complexes with 2, 4, 7,-Trinitrofluorenone: Jour., Amer. Chem. Soc., vol. 68, 1946, pp. 1727-1729.

88/ Orchin, M., and Feldman, J., The Isomerization of Dicyclohexyl: Jour. Amer. Chem. Soc., vol. 68, 1946, pp. 2737-2738.

89/ Orchin, M., Apparatus for Small-Scale Vapor-Phase Treatment of Solid Compounds: Ind. and Eng. Chem., anal. ed., vol. 17, 1945, p. 673.
Orchin, M., Aromatic Cyclodehydrogenation III. Experiments with 2-Ethylbiphenyl: Jour., Amer. Chem. Soc., vol. 68, 1946, pp. 571-2.
Orchin, M., and Friedel, R. A., Aromatic Cyclodehydrogenation IV. Experiments in the Dinaphthyl Series: Jour., Amer. Chem. Soc., vol. 68, 1946, pp. 573-4.
Orchin, M., and Reggel, L., Aromatic Cyclodehydrogenation V. A Synthesis of Fluoranthene: Jour., Amer. Chem. Soc. (In press.)

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separating the oil into neutral, acidic, and basic fractions.90/ The tar-acid fraction was composed of about the same ratio of individual tar acids as the similar fraction obtained by high-temperature carbonization. The tar-base fraction contained large quantities of aniline and the toluidines.

Only the neutral oil boiling below 175° was investigated. Low-pressure hydrogenation of this fraction facilitated the separation of the neutral fraction into aromatic, paraffin, and napthene-type compounds. After separation by selective adsorption on activated silica gel, the aromatic portion was found to contain toluene, ethylbenzene, xylenes, and a small quantity of benzene.

The paraffin-napthene portion of the neutral fraction was estimated to contain 74 percent napthenes and 26 percent paraffins. All the normal paraffin hydrocarbons from hexane to decane were present. The napthenes included cyclopentane and cyclohexane. Methylcyclohexane was the dominant component, being present to the extent of about 12.5 percent of the total paraffin-napthene fraction. More than 40 percent of the total light oil boiled above 175° C.

FOREIGN FUELS

On October 1, 1945, the technologic functions and activities of the Liquid Fuels and Lubricants and the Solid Fuels Subcommittees of the Technical Industrial Intelligence Committee were given to the Bureau of Mines.

These subcommittees were established for the purpose of investigating the German technologic developments in their respective fields. The Liquid Fuels and Lubricants Subcommittee was organized in the fall of 1944, and its first group of investigators, under the direction of Dr. W. C. Schroeder, entered German synthetic oil plants as soon as they fell into the possession of the Allied troops early in 1945. Virtually the entire contingent of investigators arrived in Europe and began conducting full-scale investigations in German and liberated territory prior to V-E day.

The Solid Fuels Subcommittee was patterned along the same lines, and its first investigators, operating under the guidance of Dr. Harry F. Yancey, entered German mines and coal-processing plants prior to the cessation of actual combat.

The members of both subcommittees were recruited from the Bureau of Mines, the Petroleum Administration for War, the universities, and private industry.

The Liquid Fuels and Lubricants group, which consisted of the following 30 authorities in the petroleum field, conducted investigations on the many phases of oil production and refining. These included all production

90/ Kaplan, E. H., Storch, H. H., and Orchin, M., Hydrogenation and Liquefaction of Coal. Part V. - Characterization of Light Oil: Bureau of Mines Tech. Paper 690, 1946, 18 pp.

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and research work on aviation gasoline, motor gasoline, Diesel and fuel oil, as well as such byproducts as synthetic lubricating oils, soaps, fats, detergents, alcohols, benzene, butadiene, and many others produced from oil, coal, and shale.

Liquid Fuels and Lubricants

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Atwell, Harold V.	The Texas Co., Beacon, N. Y.
Baldeschwieler, E. L.	Standard Oil Development Co., Bayway, N. J.
Bays, George S.	Humble Oil & Refining Co., Baytown, Tex.
Carlsmith, Leonard E.	Standard Oil Co. of Louisiana, Baton Rouge, La.
Cotton, Ernest	Gulf Oil Corp., Pittsburgh, Pa.
Doherty, Joseph D.	Bureau of Mines, Washington, D. C.
Evans, L. P.	Socony Vacuum Oil Co., Paulsboro, N. J.
Faragher, Warren F.	Houdry Process Co., Linwood, Pa.
Fraser, Donald S.	Gulf Oil Corp., Pittsburgh, Pa.
Haensel, Vladimir	Universal Oil Products Co., Riverside, Ill.
Hirst, Lester L.	Bureau of Mines, Louisiana, Mo.
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von Elbe, Guenther	Bureau of Mines, Pittsburgh, Pa.
Voss, Ernst F.	Humble Oil & Refining Co., Houston, Tex.
Weir, Horace M.	Consulting Petroleum Engineer.
Rogers, Edward J.	Bureau of Mines, Pittsburgh, Pa.
Peck, E. B.	Standard Oil Development Co., New York, N. Y.
Opal, Earl	Petroleum Administration for War, Washington, D. C.

The solid fuels group, which consisted of eight men who are leaders in the field of solid-fuel technology, was engaged in conducting investigations on research and technology of mining, coal preparation, high- and low-temperature carbonization, byproduct recovery, complete gasification, including underground gasification; domestic and industrial coal and coke combustion, gas-turbine operation, and many other related subjects.

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Solid Fuels

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Fraser, Thomas	Bureau of Mines, Washington, D. C.
Lowry, Homer H.	Carnegie Institute of Technology, Pittsburgh, Pa.
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Schmidt, Laurence D.	Bureau of Mines, Pittsburgh, Pa.
Yancey, Harry F.	Bureau of Mines, Seattle, Wash.

Although the major portion of the work of direct investigation of both teams was completed before the end of the fiscal year, it has been found necessary for two investigators to remain in Germany after the end of the year to continue with the investigation of a large volume of documents and the procuring of samples of catalysts and products and specimens of important pieces of machinery and equipment. Other investigators will be needed in the near future.

As a result of the investigations, many reports have been written, and documents supporting them have been accumulated. A few pieces of apparatus and many samples of catalysts and products have been collected. The information obtained covers the results of many years of research by leading technologists in Germany. To obtain this information independently, at least 5 years of research work by the staffs of many of the petroleum and coal research laboratories in the United States and the expenditure of many millions of dollars would be required.

The technical files and the samples of equipment, catalysts, and intermediate and end products were turned over by the Liquid Fuels and Lubricants and the Solid Fuels Subcommittees to the Bureau of Mines, which has the responsibility of indexing and digesting the files, studying the equipment and analyzing the catalysts and products, and releasing the information to the public in accordance with Presidential Orders 9568 and 9604, which direct that, so far as it may be done without prejudice to public interest, industrial information withheld from public dissemination for the purpose of national military security be released to United States industry.

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To accomplish this end, the Foreign Synthetic Liquid Fuels Division was established to process and maintain information on both liquid and solid fuels. Facilities were provided for storing and examining microfilm reels of the source documents captured in Germany. Liaison was established with the Joint Intelligence Objectives Agency, the War and Navy Departments, and the Office of Technical Services (previously known as Office of the Publication Board) in the Department of Commerce to obtain copies of all reports relating to fuels and lubricants and other subject matter for distribution to the various branches of the Bureau of Mines and for reference collection open to the general public. Considerable use has been made of this library by employees of coal, oil, and chemical companies, particularly of their research organizations.

For the benefit of those who could not conveniently make use of the library facilities, arrangements were made for the Library of Congress to provide copies of the microfilm reels. By the end of the year, 153 reels covering the entire field of interest of both subcommittees were deposited in the Library of Congress, and indexes to these reels, together with instruction for their purchase, were sent out to a mailing list compiled by the former Technical Advisory Committee of the Petroleum Administration for War.

To make the reports written by the members of the investigating teams more readily available outside of the library of the division, the reference copies relating to fuels and lubricants were reproduced on microfilm reels, which have been added to the series on deposit in the Library of Congress. Many of these reports have been previously distributed in mimeographed form prepared by the Technical Advisory Committee. Three of them have been reproduced by the Bureau of Mines and distributed to the mailing list and to visitors to the library. 91/, 92/, 93/

The availability of these reports has been announced in the Bibliography of Scientific and Industrial Reports issued weekly by the Office of Technical Services in the Department of Commerce. Every opportunity was used to publicize this bibliography and the services available in the Office of Technical Services in correspondence relating to reports and at meetings of technical societies and trade associations such as the American Institute of Mining Engineers, American Chemical Society, American Petroleum Institute, and the American Byproduct Coke Institute.

91/ Holroyd, R.; Report on Investigations by Fuels and Lubricants Teams at the I. G. Farbenindustrie A. G. Works at Leuna: C.I.O.S. Report XXXII-107, Item 30, 1945, 140 pp.; Bureau of Mines Inf. Circ. 7370, 1946, 135 pp. T.O.M. Reel 197.

92/ Hollings, H., Wintershall A. G., Lützkendorf, near Mücheln, Germany: C.I.O.S. Report XXXII-90, Item 30, 1945, 37 pp.; Bureau of Mines Inf. Circ. 7369, 1946, 21 pp. T.O.M. Reel 197.

93/ Faragher, W. F., and Horne, W. A., Manufacture and Regeneration of Catalysts at I. G. Farbenindustrie Ludwigshafen/Oppau: F.I.A.T. Report No. 422, 1945, 6 pp.; Bureau of Mines Inf. Circ. 7368, 1946, 6 pp. T.O.M. Reel 199.

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Emphasis must be placed on the fact that the reference library at its inception consisted of manuscript copies of the reports furnished by the investigators long before the machinery could be established for the reproduction of these items. Similarly, approximately 90 percent of the microfilm material was obtained prior to the establishment of the Field Intelligence Agency - Technical at Hochst (FIAT), from which a steady flow of reports and microfilms is being received.

With the active sponsorship of the American Petroleum Institute, a Film Study Group was established for the purpose of scanning the microfilm reels and abstracts, and, where warranted, to completely translate the material appearing on the microfilm reels. Reports on approximately 50 reels were completed by the Film Study Group before the end of the fiscal year. Thirty-one of these reports have been reproduced on microfilm reels, which are included among those on deposit in the Library of Congress.

Provisions for examining the samples of catalyst and intermediate and end products have been made by a special committee established by the Technical Advisory Committee of the Petroleum Administration for War. The active work of distributing the samples to the appropriate analytical groups has been conducted by the organization functioning in Pittsburgh under the supervision of Dr. H. H. Storch. Results of these analyses have just begun to come in and will also be disseminated by the microfilm route.

The activities of the division were well-received by the mining, oil, gas, and chemical industries, government agencies, and the universities. The value of this work is attested by the number of visitors making extensive use of the library facilities of the division and the prodigious amount of work donated by the Film Study Groups, which are composed of teams operating within the research organizations of some 30 leading oil companies, fuel-processing-equipment companies, and the Bureau of Mines. This is further attested by the enthusiastic cooperation of the groups in the analytical laboratories of the oil companies, the Aberdeen Proving Grounds of the U. S. Army, the Bureau of Standards, and the Bureau of Mines, who are voluntarily working on the samples of catalysts, oils, greases, and a large variety of chemicals.

Literature Translations and Abstracts

The Research and Development Division has been translating and abstracting information on synthetic liquid fuels contained in captured German documents. A preliminary report on the status of German development work on the Fischer-Tropsch and related processes was published.[94/] Translations were made of documents that present in some detail the results of research work

94/ Storch, H. H., Powell, A. R., and Atwell, H. V., Fischer-Tropsch Report No. 1 of the U. S. Government Technical Oil Mission, Sn-MC-1, T.O.M. No. 5.

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on the Fischer-Tropsch process done at the Kaiser-Wilhelm Institute für Kohlenforschung[95] and by the I. G. Farbenindustrie, A. G.

Coal-Hydrogenation Information from Captured German Documents

The information from Germany has been rather complete in providing a description of the German Synthetic Fuel Industry. The picture has been clarified quite well as regards plant and equipment operation and production problems. In addition, a great deal of factual data on unit and equipment design with, in many instances, material and heat balances, have permitted calculation of approximate thermodynamic and physical constants, heat-transfer rates, and heats of reaction. The use of this calculated information is assisting in the evaluation of necessary design data for the demonstration plant.

The mechanical engineering information gleaned from the Germans has assisted us by giving us some information on high-pressure design, particularly on handling erosive fluids. Rather complete drawings of valves, piping details, converters, and preheaters often have had more than mere descriptive value. The use of rather low alloy-content steels for exceptionally severe service has been of great metallurgical interest.

Fortunately, there is enough information to make possible a rather accurate economic study of German operating costs. These are valuable to our Research and Development Program as a base case for determining the economic impact of changes and as an orienting influence on our attempts to improve the process. These data are being organized and studied very carefully in Pittsburgh.

95/ Leva, M., Translations of German Documents on the Development of Iron Catalysts for the Fischer-Tropsch Synthesis. Part I. T.O.M. Reel 101. Doc. P. G. 21559-NID, "Report on the Middle-Pressure Synthesis with Iron Catalysts, June 1940," 51 pp.; Doc. P. G. 21574-NID, "Lecture and Discussion on Iron Catalysts for Middle-Pressure Synthesis by Dr. H. Pichler, with a preface by Director Franz Fischer, Sept. 9, 1940," 21 pp.; Doc. P. G. 21581-NID, "Recent Investigations on Iron Catalysts," 98 pp.; Doc. P. G. 21577-NID, "Research on the Behavior of Iron Catalyses when Operated with H_2-Rich Synthesis Gas," 8 pp.

Leva, M., and Atwell, H. V., Part II. T.O.M. Reel 101. Doc. P. G. 21576-NID, "Process for the Production of Hydrocarbons (Patent Application)," 10 pp.; Doc. P. G. 21578-NID, "The Middle-Pressure Synthesis with Iron Catalysts, Sept. 9, 1939," 14 pp.; Doc. P. G. 21579-NID, "Investigation of the Reaction Water Obtained from Middle-Pressure Synthesis Experiments with Iron Catalysts and Hydrogen-Rich Starting Gas, F. Weinrotter, April 30, 1941," 12 pp.; T.O.M. Reel 134, Navy 5811, Item IB-23 (first half), "Hydrocarbon Synthesis with Iron Catalysts, Leuna Works, April 5, 1940," 12 pp.; T.O.M. Reel 86, Bag 3979, Item 78, "Experiences with the Semi-Commercial Synol Research Plant ME-458 Report No. 472/44A, Leuna Works, October 10, 1944," 22 pp. Copies available in "ditto" form at Pittsburgh, Pa.

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Although many engineering details of design and operation of German coal-hydrogenation plants are contained in captured documents, there is little information on fundamental process research. Only one film (Technical Oil Mission Reel No. 145) contains a record of research in the I.G. Farben Hochdruck Laboratory. The subjects discussed in these documents concern the behavior of various coals and a multitude of "trouble-shooting" experiments designed to solve immediate production problems, but no systematic research designed to elucidate the fundamental chemistry of the process.

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